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PETROLEUM REFINING.

BY

ANDREW CAMPBELL,

MEMBER OF INSTITUTE OF PETROLEUM TECHNOLOGISTS
(MEMBER OF COUNCIL).

WITH A FOREWORD BY

SIR BOVERTON REDWOOD, BART.,
D.Sc., F.I.C., ETC.,
PAST PRESIDENT OF THE SOCIETY OF CHEMICAL INDUSTRY AND
ADVISER ON PETROLEUM TO HIS MAJESTY'S GOVERNMENT.

With 138 Illustrations, including 29 Folding Plates and
3 Diagrams, also 11 Tables.

LONDON:
CHARLES GRIFFIN AND COMPANY, LIMITED.
PHILADELPHIA: J. B. LIPPINCOTT COMPANY.
1918.

TO

JOHN TRAILL CARGILL, Esq.,

CHAIRMAN OF

THE BURMAH OIL CO. (LTD.).

385268

FOREWORD

By Sir BOVERTON REDWOOD, Bart.,

Past-President of the Society of Chemical Industry; Adviser on Petroleum to His Majesty's Government.

Although the literature of petroleum has now grown to considerable dimensions, the subject is so wide, and the need for highly specialised information on every branch so great, that it is not surprising that the ground has not been covered.

For a long time past there has been increasing need for a treatise on petroleum refining, and the deficiency has at length been met by the publication of Mr Andrew Campbell's work.

It has been cynically said that writers on technical subjects may, with few exceptions, be divided into those who are gifted with the power of lucid exposition but have little knowledge, and those who are fully informed, but are unable or unwilling to impart what they know.

The author of the present work is one of the exceptions. His long practical experience has given him unique opportunities of acquiring the fullest information in regard to the principles and practice of petroleum refining, and those who read what he has written will be satisfied that all that is worth stating has been most clearly expressed in well-chosen language, devoid of unnecessary technicalities.

The result is a treatise which will not only be indispensable to all who are engaged in the refining of petroleum, but will be of great value also to the far larger number, occupying official or commercial positions, who ought to know how the products which they are called upon to deal with are manufactured and tested.

The work has the further merit of being well illustrated, and it has been produced in the admirable manner characteristic of the firm by whom it is published.

BOVERTON REDWOOD.

PREFACE.

The fact that no book existed devoted solely to petroleum refining led me to believe that the work now presented might prove useful to the industry.

It is not historical or controversial, but merely an attempt to describe methods employed for preparing marketable products from petroleum, in both laboratory and works practice, coming within my own purview. These are the ordinary methods of refining, and do not embrace "cracking," which has been largely developed in America during the last few years.

The List of References likely to be useful to refiners in their work, and forming the Appendix (p. 223) is not claimed to be complete, and some difficulty has been experienced through the difference in spelling of authors' names in various journals. These have been arranged, as correctly as possible, in the Name Index, and I trust that any inaccuracies will be forgiven. The object of both Appendix and Name Index is to provide for refiners a wide and varied collection of the titles of papers read before and communicated to scientific societies, and published in their journals, books, etc., in a condensed form, which, it is hoped, will prove convenient and useful. Many of the original articles are in foreign languages, but the references given are in English, and will assist the searcher in deciding whether or not inquiry should be prosecuted to the source. The period covered dates from the inauguration of the Society of Chemical Industry, during which time the greatest developments in the petroleum industry have occurred.

The various authorities quoted in the text are duly acknowledged, and besides these I am indebted to :—

Mr F. Esling, A.I.C., for the descriptive sketch of experimental still; Mr E. Lawson Lomax, F.C.S., M.Sc., for preparing the notes on pitch; Miss L. G. Stevens, for assisting with proof reading and indexing; Mr R. I. Watson, for advice regarding compilation; Mr W. J. Wilson, for assistance in preparing the Appendix; Mr John Gillespie, M.I.M.E. (A. Gillespie & Son, consulting engineers, Glasgow), is entitled to an especial expression of indebtedness for preparing the Engineering Specifications (Chap. IX.), and for the original drawings of figs. 52, 53, 54, 55, 57, 58, 59, 60, 61, 62, 63, 68, 69, 133, 134, 135, 136, 137, and 138.

For descriptions of plant, etc., and for lending blocks and drawings for reproduction, my best thanks are due to the undermentioned:—

Messrs Baird & Tatlock, Glasgow, scientific instruments; Mr Herbert Barringer, London, M.Inst.C.E., water-finder; Mr Edward Cowles, Hounslow, candle machinery; Messrs J. & E. Hall, Ltd., Dartford, refrigerators; Messrs S. H. Johnson & Co., Ltd., Stratford, pyramid filter presses; Messrs J. Rhodes & Sons, Ltd., Wakefield, tin-can machinery; Messrs George Scott & Co. (London), Ltd., extractor and soda plant; Mr Henry Simmons, London, fillers and water-finder; Messrs The Steel Barrel Co., Ltd., Uxbridge, welded containers; Messrs Tintometer Co., Ltd., Salisbury, Lovibond's tintometer; Messrs E. & F. N. Spon, Ltd., kindly permitted the reference on page 140.

The patent referred to on page 118 has since been granted.

The book is dedicated to John Traill Cargill, Esq., Chairman of the Burmah Oil Co., Ltd., with which the major part of my experience has been gained, and with whose permission the work was entered upon.

That its publication should be undertaken by Messrs Charles Griffin & Co., Ltd., is most gratifying to me, as their wide experience has proved a safeguard to my first literary effort, and such collaboration has been a very pleasant experience.

In conclusion, I desire to tender my grateful thanks to Sir Boverton Redwood, Bart., for his encouragement in the preparation of the manuscript, for examining it and permitting quotations from his *Treatise on Petroleum*, and for so kindly adding his generous Foreword.

ANDREW CAMPBELL.

BECKENHAM, KENT,
April 1918.

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PETROLEUM REFINING.

CHAPTER I.

EXAMINATION FOR YIELDS OF MARKETABLE PRODUCTS.

The first step to be taken by an oil refiner in considering a refining problem is the examination of the crude material with a view to determining (1) quality and quantity of marketable products, and (2) the necessary plant required therefor.

In these connections the points for determination are :—

1. Estimation of dirt and water, if any.
2. Specific gravity.
3. Flashing-point.
4. Setting or solidifying point.
5. Estimation of benzine, kerosene, and heavy oil by Engler's method.
6. Estimation of sulphur content, if any, and calorific value.
7. Examination of 5 per cent. fractions obtained by distillation of not less than 5 gallons.

1. **Estimation of Dirt and Water.**—This is best accomplished in a Sutherland bulb (fig. 1).

A measured quantity of the oil is placed in the bulb, which is kept in a water-bath maintained at a temperature about 180° F. until complete separation is effected. The amount of solid matter and water settled out may then be observed in the graduated tube fused to the bottom of the bulb, and the result calculated to percentage.

If the oil is volatile, it may be necessary to tie down the stopper to prevent loss by evaporation, and if of a heavy viscous nature, the sample should be mixed with a measured quantity of kerosene to effect more rapid separation.

For all the following tests the oil must be free from the presence of dirt and water, if any should be present, in original samples.

2. **Specific Gravity.**—With the exception of pitch and solid paraffin, all petroleum products are liquid or semi-liquid. The specific gravity of pitch should be determined by the method described on p. 85. The specific gravity of wax (solid paraffin) may

also be determined by the ordinary method for solids lighter than water, or, at a temperature at which it is liquid, by the hydrometer, but for all practical purposes the specific gravity of solid paraffin may be taken as 0·8 compared with water 1·0 at 60° F. The specific gravity of all other products may be determined by one or other of the several methods used for liquids, but the instrument generally employed is the hydrometer. Specific gravity reported without reference to the temperature of determination is understood to be at the normal temperature, 60° F. It is not necessary, and not always convenient, to determine specific gravity of oils at that temperature, and if determined at a higher or lower temperature a correction is made for coefficient of expansion, as explained later. If the temperature of determination is higher, the figure equal to the number of degrees above 60° F. is added to, and if lower deducted from, the actual specific gravity determined.

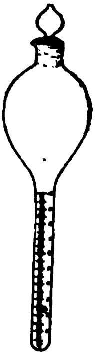

Fig. 1.—Sutherland bulb.

As regards *coefficient of expansion*, Sir Boverton Redwood [1] says :—"The following corrections for each 1° F. should in practice be made. For products lighter than kerosene, 0·00040 to 0·00048 ; for kerosene, 0·00040 ; for gas oils, 0·00036 ; for lubricating oils, 0·00034."

The temperature at which the specific gravity of heavy oils containing paraffin, or of highly viscous oils, is determined should only be high enough to allow the operation to be performed without the oil becoming too viscous, or the solid paraffin, should it contain any, crystallising.

Specific gravity, or density of a liquid, or solid body, is its weight, as compared with that of an equal volume of pure water, at 60° F.=15·5° C.

The methods for the determination of specific gravity are :—(1) For solids, the chemical balance ; and (2) for liquids, the chemical balance and the hydrometer. The latter is the instrument generally used throughout the petroleum industry, both in laboratory and refinery practice. It is usual to preserve standard hydrometers and thermometers for checking the inaccuracies of those issued to workmen engaged throughout the refinery, and scientific instrument-makers supply these, with a certificate of accuracy granted by the National Physical Laboratory after the instruments have been tested.

Hydrometers may be checked by immersing them in a liquid the specific gravity of which has been determined by the chemical balance.

For the determination of the specific gravity of a solid body, which is insoluble in, but heavier than, water, it is first weighed in air in the ordinary manner, and then weighed in distilled water at 60° F., the normal temperature. When the substance is lighter than water, after its weight is determined in the ordinary manner, it is attached to a weight sufficiently heavy to sink it in the water. Should the substance, the specific gravity of which has to be determined, be soluble in water, a liquid in which it is insoluble, and of known specific gravity, is employed. If the substance is in a powdered condition it is weighed in a specific gravity bottle, which is afterwards filled with distilled water and again weighed.

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[1] *Treatise on Petroleum*, 3rd Ed., vol. i. p. 218.

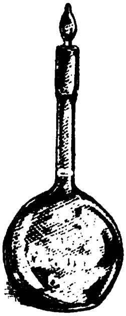

FIG. 2.—Specific gravity bottle.

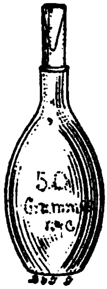

FIG. 3.—Specific gravity bottle.

Liquids are weighed in a bottle. Fig. 2 shows a form which is used for all liquids. It is graduated to its capacity by a mark on the neck, and is provided with a perfectly ground, blown glass stopper. Another form of bottle used for non-volatile liquids is that illustrated by fig. 3. The stopper of this is perforated so that it may be filled completely.

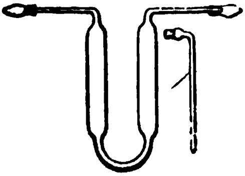

FIG. 4.—Pyknometer.

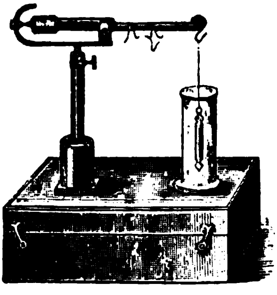

FIG. 5.—Westphal balance.

The pyknometer, or Sprengel tube (fig. 4), is also used for the determination of specific gravity, and under certain conditions is more convenient than the bottle. The latter, however, is the vessel employed when specific gravities are checked on the balance. It is unnecessary to refer further to the former.

Special balances are made for observing the specific gravity of liquids. The Westphal balance is one of this type (fig. 5). Before being used it is adjusted to be in equilibrium when the plummet is in the air. The liquid to be tested is placed in a glass jar and the plummet suspended in it. If water is the liquid, the largest and heaviest rider must be placed on the hook, from which the plummet hangs, to restore equilibrium. The balance beam is divided into ten equal parts, indicated by notches, and rider weights are provided, each one-tenth the weight of the next largest.

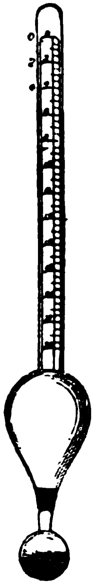

FIG. 6.—Ordinary hydrometer.

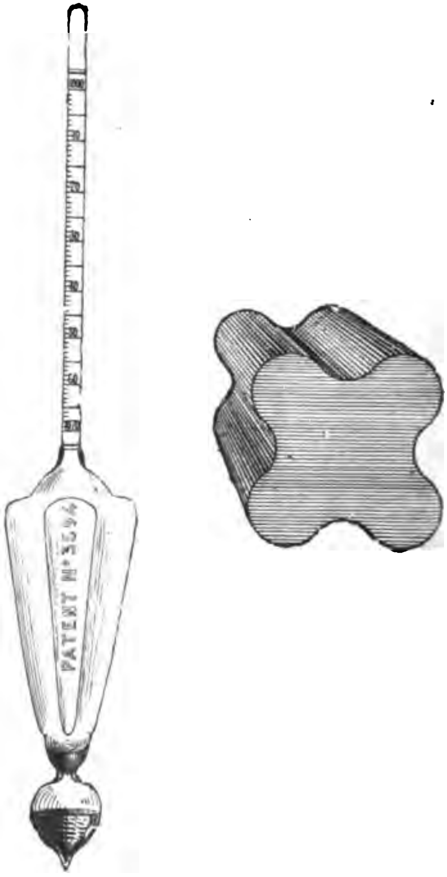

FIG. 7.—Stronger hydrometer.

The Sartorius specific gravity balance is another instrument of this type.

The specific gravity balance is very convenient when conducting a laboratory distillation in small fractions.

The foregoing brief general description of the apparatus and methods used for determination of specific gravity by the chemical balance is only given here for the information of those readers unfamiliar with such processes. Many works on chemical analysis contain the full definitions and explanations of the subject, to which reference may be made.

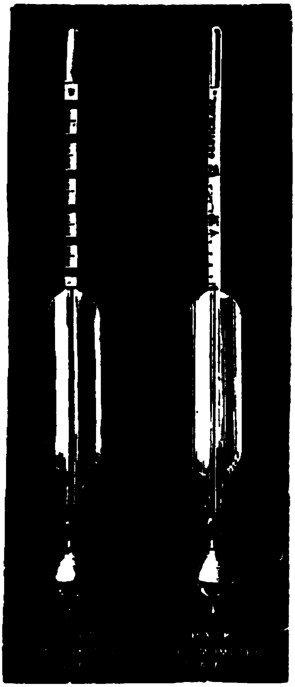
FIG. 8.—Thermo-Hydrometer.

Fig. 6 illustrates the ordinary hydrometer. When it is immersed in a liquid it sinks, the specific gravity being indicated by the number on the stem level with the surface of the liquid. Hydrometers are made in series, say from ·650 to ·700, ·700 to ·750, and so on. Fig. 7 shows a stronger form of instrument.

Hydrometers are also made with a thermometer fixed inside the bulb and stem, enabling the temperature and specific gravity to be observed at exactly the same moment without the use of a separate instrument to ascertain the temperature (fig. 8). The common type of vessel for containing the liquid to be tested is the hydrometer jar (fig. 9).

Thermometers graduated both in the Fahrenheit and Centigrade scales are employed. In the refinery the former has become customary, while in the laboratory flash-points, melting-points, and setting-points are usually determined by the Fahrenheit, and fractionations conducted by the Centigrade, scale. The one may be converted to the other by making the following calculations :—

°F to °C.—

$$\frac{°F.-32}{9} \times 5 = °C.$$

°C. to °F.—

$$\frac{°C. \times 9}{5} + 32 = °F.$$

Tables for the comparison of the two scales will be found in many books of reference.

FIG. 9.—Hydrometer jar.

Hydrometers are also made in degrees Baumé, but these are not employed to any extent by British refiners. It is necessary to check cargoes reported in that scale, and this may be done by the following calculations :—

60° F.— $$\frac{140}{°B.+130}=\text{sp. gr.}$$

60° F.— $$\frac{140}{\text{sp. gr.}}-130=\text{degrees Baumé.}$$

Another scale for the graduation of hydrometers is that known as the "Twaddell." It has become the practice in refineries to express the strength of acid and soda solutions in terms of that scale. When necessary these may be converted into specific gravity by multiplying the degrees Twaddell by 5, adding 1000, and dividing the result by 1000.[1]

3. **Flashing-point** is the lowest temperature at which an oil, when heated at a determined rate, gives off vapour which will "flash" when a light is applied.

For the examination of crude petroleum, or fractions distilled therefrom, the "close" type of apparatus is employed. A variety of instruments have been designed for the close test, but the following may be accepted as standards :—

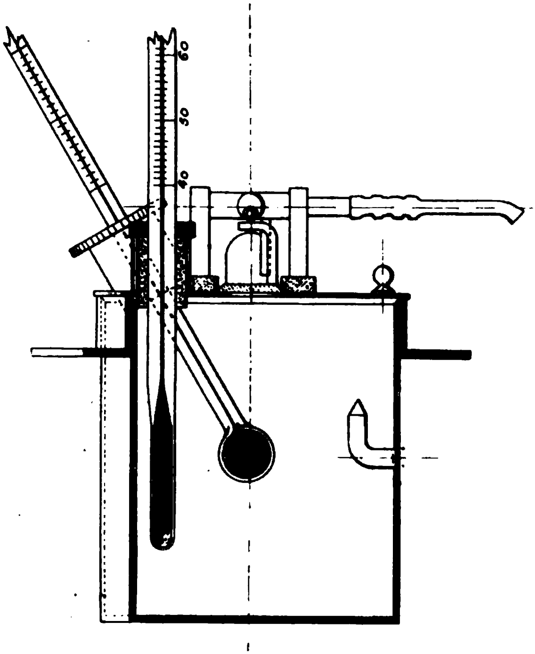

FIG. 10.—Abel-Pensky apparatus. Showing position of two thermometers for testing low-flash oils solid at ordinary temperatures.

(1) The Abel standard flash-point apparatus (fig. 13).

(2) The Abel-Pensky apparatus (Redwood modification), with additional side thermometer (fig. 10).

(3) The Gray flash-point apparatus (figs. 11 and 12).

The first instrument is employed for all oils liquid at ordinary temperatures flashing within the range of temperatures specified in the directions for its use, which must be carefully adhered to. It may also be used for flash-points up to 150° F. by increasing the temperature of the water in the heating vessel.

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[1] A useful volume containing all such data is Redwood and Eastlake's *Petroleum Technologist's Pocket Book* (C. Griffin & Co., Ltd.).

The second instrument is used for testing crude oils, which, though *not* liquid at ordinary temperatures, give off inflammable vapour.

The Gray tester is for "heavy" oils, and may be used for all oils flashing over 150° F.

The "open" flashing-point may be determined in the cup of any of the above instruments from which the cover has been removed and arrangement made for the suspension of the thermometer.

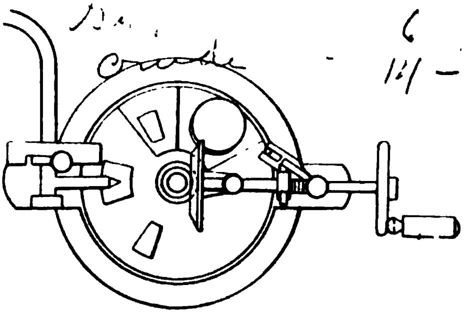
The "fire" test is the temperature at which the vapours from an oil heated in an open vessel will, when a light is applied at regular intervals, "fire" and continue to burn.

The following quotation[1] explains the application of these two tests and their value for ascertaining the homogeneous character of an oil when considered in conjunction with the close-test results:—

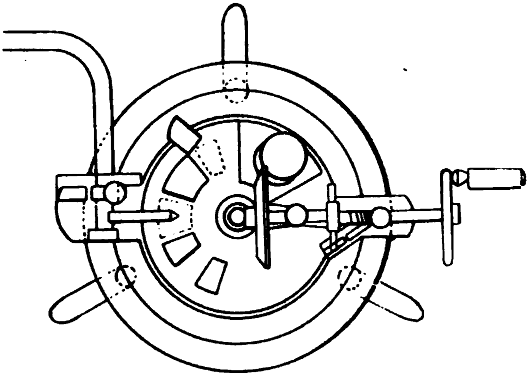
"These tests, in conjunction with the close test, give some idea as to the homogeneous character of the sample under examination.

crude

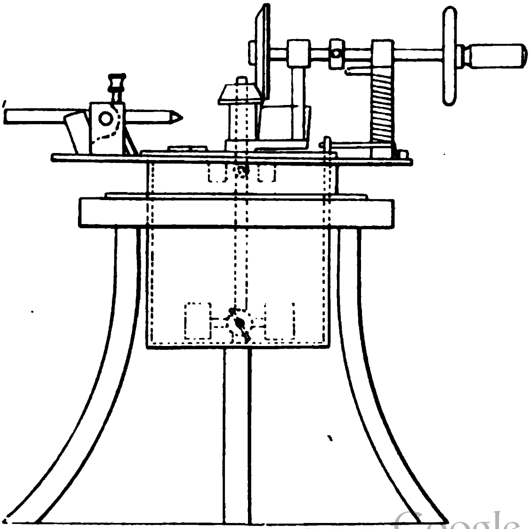
C 58
11 – 35



Plan, slide open.



Plan, slide closed.



Elevation.

FIG. 11.—Gray's flash-point apparatus.

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[1] *Laboratory Book of Mineral Oil Testing*, Jas. A. Hicks (C. Griffin & Co., Ltd.).

"It is possible, for instance, to detect the presence of a small percentage of a light oil in a lubricant by the wide range covered by the three tests. Very often, in such cases, if the proportion of light oil be not too great, by allowing the cupful to cool after the fire test is taken, and then repeating the whole experiment, a very great difference between the first and second close tests, a smaller difference between the open tests, and a practical agreement between the fire tests will confirm such an inference. But it must always be remembered that this treatment will invariably cause *some* alteration in the lower figures.

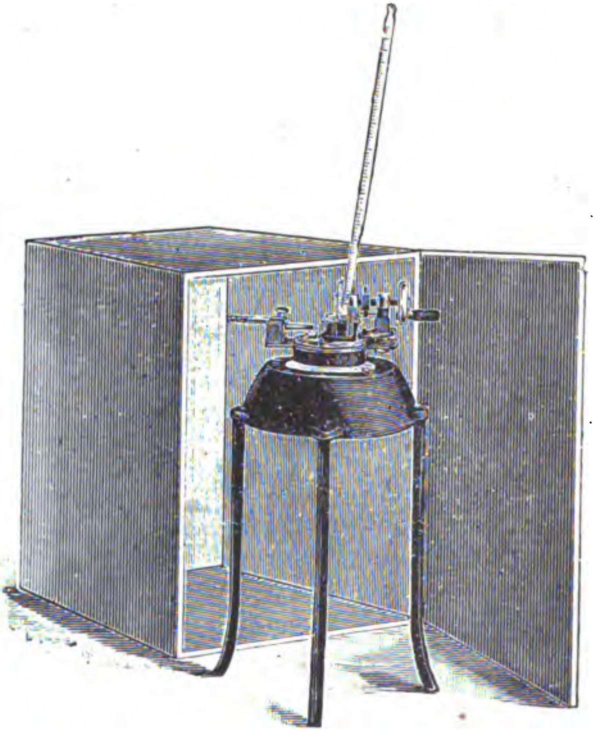

FIG. 12.—Gray's flash-point apparatus.

"The usual way of taking the open and fire tests is to continue the heating when the close test has been ascertained, and, removing the lid of the cup, to hang the thermometer from a retort stand with its bulb immersed about a quarter of an inch below the surface of the oil. Where absolute accuracy is needed, a fresh portion of oil should be taken, and the open test and fire test made with the lid of the cup removed throughout the experiment, and the level of the oil at the temperatures of the results not much more than an eighth of an inch below the top of the cup. At every 2° F. a test-flame of gas, $\frac{3}{16}$ of an inch in diameter, is passed across the surface of the oil slightly below the level of the cup, taking care that it does not come in contact with the oil. The heating remains at the same rate of 10° F. a minute. That point at which a flicker of flame covers the *whole* of the surface is noted as the open flash, and the fire test is the temperature at which the oil vapours continue to burn until the next application of the flames is due.

"The two higher tests may also be performed by heating the oil in a porcelain crucible of about two inches diameter in a sand-bath. The crucible is filled to about a quarter of an inch from the top, and the level of the sand and oil should be in the same plane.

"In the absence of coal-gas for the test-flame a useful substitute can be provided by hydrogen or air which has been passed through a Woulff's bottle containing cotton-wool saturated with light petroleum spirit."

The following extract with reference to the designs and methods of using the Abel and Abel-Pensky instruments is reproduced here for ready reference from the Third Edition of Sir Boverton Redwood's *Treatise on Petroleum*, vol. ii. :—

"In 1875 the Government, with the concurrence of the Metropolitan Board of Works and of the Petroleum Association, requested Sir Frederick Abel to experimentally investigate the matter, with a view to placing it on a satisfactory basis. The questions referred were as follows :—

"(1) Whether the method of testing petroleum as prescribed in Schedule 1 of the Petroleum Act, 1871 (34 & 35 Vict., c. 105), is such as uniformly to ensure reliable and satisfactory results.

"(2) If not, what alterations in the method of testing petroleum should be adopted to secure such results, due regard being had to the fact that the testing must, in many instances, be carried out by persons who have had comparatively little experience in conducting delicate experiments.

"(3) Assuming it to be, in your opinion, desirable to obtain a 'flashing-test' for petroleum, whether the present 'flashing-point' of 100° F. (or its equivalent under any modified method of testing which you may propose) is, in your judgment, calculated to afford adequate protection to the public, without unduly interfering with or restricting the trade; if not, what alteration in this respect should be made.

"After a prolonged and exhaustive experimental inquiry, in which Dr Kellner rendered valuable assistance, Sir Frederick Abel presented to the Secretary of State an elaborate report, dated 12th August 1876, wherein the questions enumerated were thus answered :—

"(1) The method of testing petroleum as prescribed in Schedule 1 of the Petroleum Act, 1871 (34 & 35 Vict., c. 105), is not of a nature 'uniformly to ensure reliable and satisfactory results.'

"(2) A method of testing petroleum has been elaborated for adoption in place of that prescribed by the Petroleum Act, 1871, due regard having been had to the fact 'that the testing must, in many instances, be carried out by persons who have had comparatively little experience in conducting delicate experiments.' This method, while resembling in its general nature the one hitherto used, is free from the defects inherent in the latter, and is so arranged that it can be carried out, with the certainty of furnishing uniform and precise results, by persons possessing no special knowledge or skill in manipulation. With ordinary attention in the first instance to simple instructions, different operators cannot fail to obtain concordant results with it, and it is so nearly automatic in its nature that it is not, like the present method of testing, susceptible of manipulation so as to furnish different results at the will of the operator.

"(3) There are not, in my judgment, any well-established grounds for considering that the present flashing-point of 100° F. is not 'calculated to afford adequate protection to the public.'

"(4) With the employment of the new test, a minimum flashing-point should therefore be adopted, which is equivalent, or as nearly as possible so, to the flashing-point of 100° F., as furnished by the present test.

"In the determination of the equivalent flashing-point, it was obviously necessary to deal with the conflicting views already referred to as to the proper mode of conducting the test with the open-cup instrument. The author was enabled to assist Sir Frederick Abel in the conduct of this portion of the inquiry, but before the test standard was even provisionally fixed, Mr T. W. Keates, as representing the Metropolitan Board of Works, and Mr John Calderwood, on behalf of the Scottish Mineral Oil Association, were also consulted. Eventually, as the result of the joint experiments, it was ascertained that the difference between the flashing-points obtained with the open-cup instrument and those furnished by the Abel apparatus usually ranged from 25° to 29° F. Adopting the mean difference of 27°, the new standard was accordingly fixed at 73° F. The author then applied the two tests to 1000 samples of American kerosene, the first 968 samples consisting of the ordinary oil of commerce, and the remaining 32 of "water-white" oil. The following were the results obtained :—

92 samples showed a difference between the two tests of 25°
208 ,, ,, ,, ,, ,, ,, 26°
225 ,, ,, ,, ,, ,, ,, 27°
281 ,, ,, ,, ,, ,, ,, 28°
162 ,, ,, ,, ,, ,, ,, 29°
———
968

"On the other hand, the majority of the last 32 samples gave smaller differences, as follows :—

9 samples showed a difference between the two tests of 20°
1 sample ,, ,, ,, ,, ,, 21°
9 samples ,, ,, ,, ,, ,, 22°
1 sample ,, ,, ,, ,, ,, 23°
4 samples ,, ,, ,, ,, ,, 24°
8 ,, ,, ,, ,, ,, ,, 25°
—
32

"These, however, all consisted not of ordinary petroleum oil, but of the special kind which is known in the trade under the name of "water-white oil," and, therefore, the exceptional results afforded by them do not affect the question at issue, and are of interest only as showing that samples may be selected or specially prepared having flashing-points by the two systems more closely approximating than those of the ordinary petroleum oil of commerce. This "water-white oil," as is well understood, possesses the distinctive feature of low specific gravity in addition to that of high flashing-point, being, in fact, produced at a considerably enhanced cost, by rejecting, in the process of distilling the crude oil, an unusually large proportion of the heavier as well as the lighter hydrocarbons, and doubtless this accounts for the smaller difference between the two tests.

"Before the new test was legalised, the author proceeded (in 1877) to New York for the purpose of submitting the instrument to the inspectors appointed by the New York Produce Exchange, and arranging for its use in testing kerosene intended for shipment to this country.

"**The Abel Test.**—The result of Sir Frederick's investigation was the legalisation, on 11th August 1879, of the "Abel test." The instrument and its use are thus described in the Petroleum Act of that date :—

“FIRST SCHEDULE.

“MODE OF TESTING PETROLEUM SO AS TO ASCERTAIN THE TEMPERATURE AT WHICH IT WILL GIVE OFF INFLAMMABLE VAPOUR.

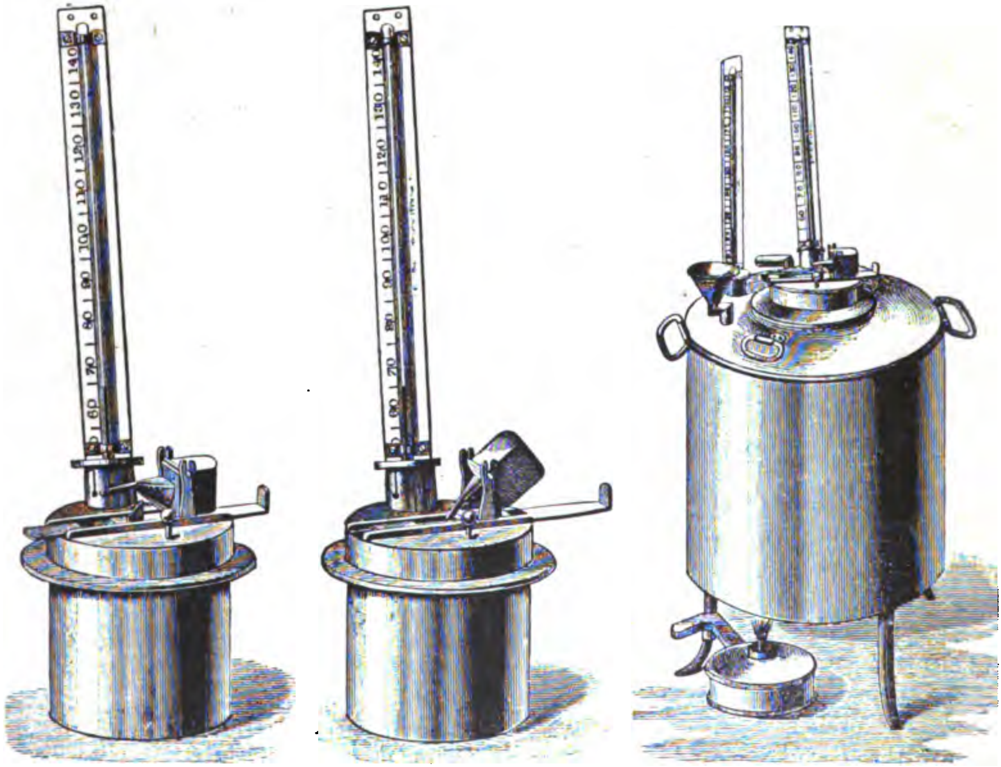

FIG. 13.—Abel standard flash-point apparatus.

“**Specification of the Test Apparatus** (fig. 13).—The following is a description of the details of the apparatus :—

“The oil-cup consists of a cylindrical vessel 2 inches diameter, $2\frac{2}{10}$ inches height (internal), with outward projecting rim $\frac{5}{10}$ inch wide, $\frac{3}{8}$ inch from the top, and $1\frac{7}{8}$ inches from the bottom of the cup. It is made of gun-metal or brass (17 B.W.G.) tinned inside. A bracket, consisting of a short stout piece of wire bent upwards, and terminating in a point, is fixed to the inside of the cup to serve as a gauge. The distance of the point from the bottom of the cup is $1\frac{1}{2}$ inches. The cup is provided with a close-fitting overlapping cover made of brass (22 B.W.G.), which carries the thermometer and test-lamp. The latter is suspended from two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, the mouth of which is 1/16 inch diameter. The socket which is to hold the thermometer is fixed at such an angle and its length is so adjusted that the bulb of the thermometer, when inserted to its full depth, shall be 1½ inches below the centre of the lid.

" The cover is provided with three square holes—one in the centre, 5/10 inch by 4/10 inch, and two smaller ones 3/10 inch by 2/10 inch close to the sides and opposite each other. These three holes may be closed and uncovered by means of a slide moving in grooves, and having perforations corresponding to those on the lid.

" In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted in such a way as to bring the end of the spout just below the surface of the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position.

" Upon the cover, in front of and in line with the mouth of the lamp, is fixed a white bead, the dimensions of which represent the size of the test-flame to be used.

" The bath or heating-vessel consists of two flat-bottomed copper cylinders (24 B.W.G.)—an inner one of 3 inches diameter and 2½ inches in height, and an outer one of 5½ inches diameter and 5¾ inches in height; they are soldered to a circular copper plate (20 B.W.G.) perforated in the centre, which forms the top of the bath, in such a manner as to enclose the space between the two cylinders, but leaving access to the inner cylinder. The top of the bath projects both outwards and inwards about ⅜ inch; that is, its diameter is about ⅜ inch greater than the body of the bath, while the diameter of the circular opening in the centre is about the same amount less than that of the inner copper cylinder. To the inner projection of the top is fastened, by six small screws, a flat ring of ebonite, the screws being sunk below the surface of the ebonite to avoid metallic contact between the bath and the oil-cup. The exact distance between the sides and bottom of the bath and of the oil-cup is ½ inch. A split socket similar to that on the cover of the oil-cup, but set at a right angle, allows a thermometer to be inserted into the space between the two cylinders. The bath is further provided with a funnel, an overflow-pipe, and two loop handles.

" The bath rests upon a cast-iron tripod stand, to the ring of which is attached a copper cylinder or jacket (24 B.W.G.) flanged at the top, and of such dimensions that the bath, while firmly resting on the iron ring, just touches with its projecting top the inwardturned flange. The diameter of this outer jacket is 6½ inches. One of the three legs of the stand serves as a support for the spirit-lamp attached to it by means of a small swing bracket. The distance of the wick-holder from the bottom of the bath is 1 inch.

"Two thermometers are provided with the apparatus—the one for ascertaining the temperature of the bath, the other for determining the flashing-point. The thermometer for ascertaining the temperature of the water has a long bulb and a space at the top. Its range is from about 90° to 190° F. The scale (in degrees of Fahrenheit) is marked on an ivory back fastened to the tube in the usual way. It is fitted with a metal collar, fitting the socket, and the part of the tube below the scale should have a length of about 3½ inches, measured from the lower end of the scale to the end of the bulb. The thermometer for ascertaining the temperature of the oil is fitted with collar and ivory scale in a similar manner to the one described. It has a round bulb, a space at the top; and ranges from about 55° F. to 150° F.; it measures, from end of ivory back to bulb, 2¼ inches."

Note.—A model apparatus is deposited at the Weights and Measures Department of the Board of Trade.

"DIRECTIONS FOR APPLYING THE FLASHING-TEST.

"(1) The test-apparatus is to be placed for use in a position where it is not exposed to currents of air or draughts.

"(2) The heating vessel or water-bath is filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test is to be 130° F., and this is attained in the first instance either by mixing hot and cold water in the bath, or in a vessel from which the bath is filled, until the thermometer which is provided for testing the temperature of the water gives the proper indication; or by heating the water with the spirit-lamp (which is attached to the stand of the apparatus) until the required temperature is indicated.

"If the water has been heated too highly, it is easily reduced to 130° by pouring in cold water little by little (to replace a portion of the warm water) until the thermometer gives the proper reading.

"When a test has been completed, this water-bath is again raised to 130° by placing the lamp underneath, and the result is readily obtained while the petroleum-cup is being emptied, cooled, and refilled with a fresh sample to be tested. The lamp is then turned on its swivel from under the apparatus, and the next test is proceeded with.

"(3) The test-lamp is prepared for use by fitting it with a piece of flat plaited candle-wick, and filling it with colza or rape oil up to the lower edge of the opening of the spout or wick-tube. The lamp is trimmed so that when lighted it gives a flame of about 0·15 of an inch in diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil-cup,[1] is readily maintained by simple manipulation from time to time with a small wire trimmer.

"When gas is available it may be conveniently used in place of the little oil-lamp, and for this purpose a test-flame arrangement for use with gas may be substituted for the lamp.

"(4) The bath having been raised to the proper temperature, the oil to be tested is introduced into the petroleum-cup, being poured in slowly until the level of the liquid just reaches the point of the gauge which is fixed in the cup. In warm weather the temperature of the room in which the samples to be tested have been kept should be observed in the first instance, and if it exceeds 65°, the samples to be tested should be cooled down (to about 60°) by immersing the bottles containing them in cold water, or by any other convenient method. The lid of the cup, with the slide closed, is then put on, and the cup is placed into the bath or heating vessel. The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid, and its position is not, under any circumstances, to be altered. When the cup has been placed in the proper position, the scale of the thermometer faces the operator.

"(5) The test-lamp is then placed in position upon the lid of the cup, the lead-line or pendulum, which has been fixed in a convenient position in front of the operator, is set in motion, and the rise of the thermometer in the petroleum-cup is watched. When the temperature has reached about 66°, the operation of testing is to be commenced, the test-flame being applied once for every rise of one degree in the following manner:—

"The slide is slowly drawn open while the pendulum performs three oscillations, and is closed during the fourth oscillation.

"*Note.*—If it is desired to employ the test apparatus to determine the flashing-point of oils of very low volatility, the mode of proceeding is to be modified as follows:—

"The air-chamber which surrounds the cup is filled with cold water to a depth of 1½ inches, and the heating vessel or water-bath is filled as usual, but also with cold water. The lamp is then placed under the apparatus, and kept there during the entire operation. If a very heavy oil is being dealt with, the operation may be commenced with water previously heated to 120°, instead of with cold water.

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[1] "It was at first intended merely to specify the size of the flame, but as it was found difficult to judge of the diameter, the bead was added at the suggestion of the author."

"The pendulum described in (5) measures 24 inches from the point of suspension to the centre of gravity of the weight. The author has found it convenient to employ a synchronised pendulum clock, or a metronome, when making a large number of tests.

"The Abel instruments employed in the United Kingdom by the inspectors appointed in accordance with the provisions of the Petroleum Acts are standardised by the Weights and Measures Department of the Board of Trade.

"The Petroleum Act, 1879, gives no specific directions applicable to the testing of indiarubber solution, and to meet this deficiency the first schedule of the Inflammable Liquids Bill, 1891, contained the following addition to the instructions for the use of the test apparatus :—

"If the flashing-test has to be applied to substances of a viscous or semi-solid nature which cannot be poured (such as solutions of indiarubber in mineral naphtha), the mode of proceeding is as follows :—About a tablespoonful of the substance to be tested is placed in the cup, and the cover is put on. The air-chamber in the water-bath is filled with water to a depth of 1½ inches, and the temperature of the water-bath is raised to 76° (for which purpose the scale of the thermometer to be used in the water-bath should range from 60° to 180°).

"The cup is then put into the bath, and the temperature of the water-bath maintained at 76° throughout the test. After the lapse of fifteen minutes the test-flame is to be applied. If no flash occurs, the heating is continued for another fifteen minutes ; and the test-flame again applied, and so on until a flash takes place, or the temperature in the cup has reached 75° ; [if a flash occurs at or] [1] below 72°, the substance under examination has an observed flashing-point of less than 73°. The temperature at which a flash occurs is the observed flashing-point of the substance, and, subject to correction for atmospheric pressure as hereinbefore described, is the true flashing-point.

"Although some paints containing petroleum, such as anti-fouling and anti-corrosion compositions applied to ships' bottoms, and some compounds used as liquid metal polish, are neither viscous nor semi-solid and can be poured, their flashing-points cannot be satisfactorily determined in the manner prescribed for the testing of petroleum owing to their being insufficiently fluid to admit of the formation of convection-currents. In other words, the heat applied by means of the water-bath and air-jacket does not circulate freely through the contents of the oil-cup, with the result that the part of the liquid undergoing the test which is in contact with the walls of the cup

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[1] The words in square brackets have apparently been inadvertently omitted.

acquires a much higher temperature than is indicated by the thermometer, and a flashing-point may be thus recorded which is far below the true flashing-point. For the efficient testing of such liquids a stirrer in the oil-cup is needed."

The following semi-official memorandum on this subject was published in *The Times* of 17th May 1904 :—

"**Petroleum Acts.**—It has recently come to our knowledge that attempts are being made by officers of local authorities to apply the legal test for petroleum to samples of liquid metal polish, and we have satisfied ourselves by experiment that the results thus obtained are sometimes entirely misleading, as they do not represent the temperature of the portion of the liquid from which inflammable vapour is being evolved.

"In carrying out the test prescribed by the Petroleum Act, 1879, the sample under examination is slowly heated in a closed cup, and the temperature is indicated by a thermometer, the bulb of which is immersed in the liquid in the centre of the vessel. In these circumstances, the heat communicated to the sample through the walls of the cup creates, in such a liquid as petroleum, convection-currents, and through the circulation thus set up the temperature of the contents of the cup is equalised, and the thermometer correctly indicates the temperature at which inflammable vapour is evolved by the liquid.

"On the other hand, if the sample contains solid matter in suspension, as is the case with the liquid metal polish in question, the formation of convection-currents is interfered with, and the surface of the liquid from which inflammable vapour is evolved acquires a higher temperature than that of the portion in contact with the bulb of the thermometer. Thus, as we have ascertained experimentally, the thermometer may indicate a temperature of 59° F., when the temperature of the surface is 83° F., and a sample may be erroneously reported as having a flash-point below the legal limit of 73° F., when the true flash-point is far above the limit.

"No doubt this would have been provided for when the Act was passed if, at that time, the need for applying the test to such substances had been foreseen, but it was not until judgment in the case of The London County Council *v.* Holzapfels Compositions Company, Limited, was given in 1899 that mixtures containing petroleum were held to be petroleum within the meaning of the Acts.

"In our *Handbook on Petroleum*, published in 1901, we referred, on p. 90, to the necessity for a stirrer in the oil-cup when the test specified in the Petroleum Act is employed for the testing of paints and other substances containing petroleum, and when opportunity occurs for a revision of the law this addition will doubtless be legalised.

"We are, however, of opinion that, in the meantime, authorities charged with the administration of the Petroleum Acts should be made aware of the circumstances we have referred to, and that testing officers should take steps to ascertain the true flash-point of samples of liquid metal polish or other substances which are not thoroughly liquid, and, therefore, cannot be satisfactorily tested in precise accordance with the directions given in the schedule to the Act. In many instances it may be possible to obtain a sample of the petroleum used in the substance, or the solid matter present in the sample can be removed by straining or filtration, care being taken to avoid loss of the more volatile constituents by evaporation, when the separated liquid can be tested in the prescribed manner. The liquid should not, however, be separated by distillation, as this operation may yield a distillate of lower flash-point than that of the petroleum with which the mixture was made. For guidance in determining whether there has been any infraction of the law, the sample may also be tested in an apparatus provided with an efficient stirrer. In any case of doubt as to the true flash-point of the material, we would suggest that reference should be made to His Majesty's Inspector of Explosives, at the Home Office, who will be prepared to give advice as to the course which should be adopted.

"J. H. THOMSON, Captain,
"H.M. Chief Inspector of Explosives.

"BOVERTON REDWOOD,
"Adviser on Petroleum to the Home Office."

"30th April 1904."

By an Order in Council, dated 7th May 1907, it was directed that certain portions of the Petroleum Acts, 1871 to 1881, shall apply to mixtures of petroleum, and the following instructions for testing were given :—

"DIRECTIONS FOR TESTING PETROLEUM MIXTURES.

"(1) **Liquid Mixtures.**—Where the petroleum mixture is wholly liquid, flows quite freely, and does not contain any sediment or thickening ingredient, such mixture shall be tested in the manner set forth in Schedule One to the Petroleum Act, 1879.

"(2) **Viscous and Sedimentary Mixtures.**—Where the petroleum mixture contains an undissolved sediment, as in the case of some metal polishes, which can be separated by filtration or by settlement and decantation, the sediment may be so separated, and the decanted liquid may be tested in the manner set forth in Schedule One to the Petroleum Act, 1879.

"In carrying out such separation, care must be taken to minimise the evaporation of the petroleum. The separation of the sediment must not be effected by distillation.

"Where the petroleum mixture is such that sediment cannot be separated by the aforementioned means, or where it is of a viscous nature, as in the case of indiarubber solution, quick-drying paints, etc., such mixture shall be tested in the apparatus modified as shown in the drawing hereto (fig. 14). This apparatus differs from that prescribed in Schedule One to the Petroleum Act, 1879, only in the addition of a stirrer to equalise the temperature throughout the sample under test.

"In carrying out the test of a viscous petroleum mixture, this stirrer shall be constantly revolved at a slow speed, except when applying the test-flame, with the fingers, the direction of revolution being that of the hand of a clock.

"With the exception of the use of a stirrer, the manner of carrying out the test shall be that set forth in Schedule One to the Petroleum Act, 1879.

"The stirrer may be removed by grasping the spindle just above the blades with the finger and thumb, and unscrewing the upper sheath. The opening in the lid, through which the stirrer passes, may then be closed by a plug provided for the purpose.

"When this has been done, the apparatus shall be deemed to comply with the specification set forth in Schedule One of the Petroleum Act, 1879, and may be used for testing ordinary petroleum or solid petroleum mixtures.

"A model of the aforementioned apparatus will be deposited with the Board of Trade, and the provisions of section 3 of the Petroleum Act, 1879, in regard to verification and stamping, shall apply also to such apparatus as though it were the apparatus prescribed by the said Act.

"For the purpose of carrying out such verification the stirrer shall be removed and the opening plugged as hereinbefore directed. The apparatus shall then be tested with ordinary petroleum. The stirrer shall be verified by comparison of measurements.

"(3) **Solid Petroleum Mixtures.**—Where the petroleum mixture is solid, as in the case of naphtha soaps, etc., the apparatus to be used for the test shall be that prescribed in Schedule One of the Petroleum Act, 1879.

"The method of carrying out the test of such solid mixture shall be as follows:—

"The solid mixture must be cut into cylinders 1½ inches long and

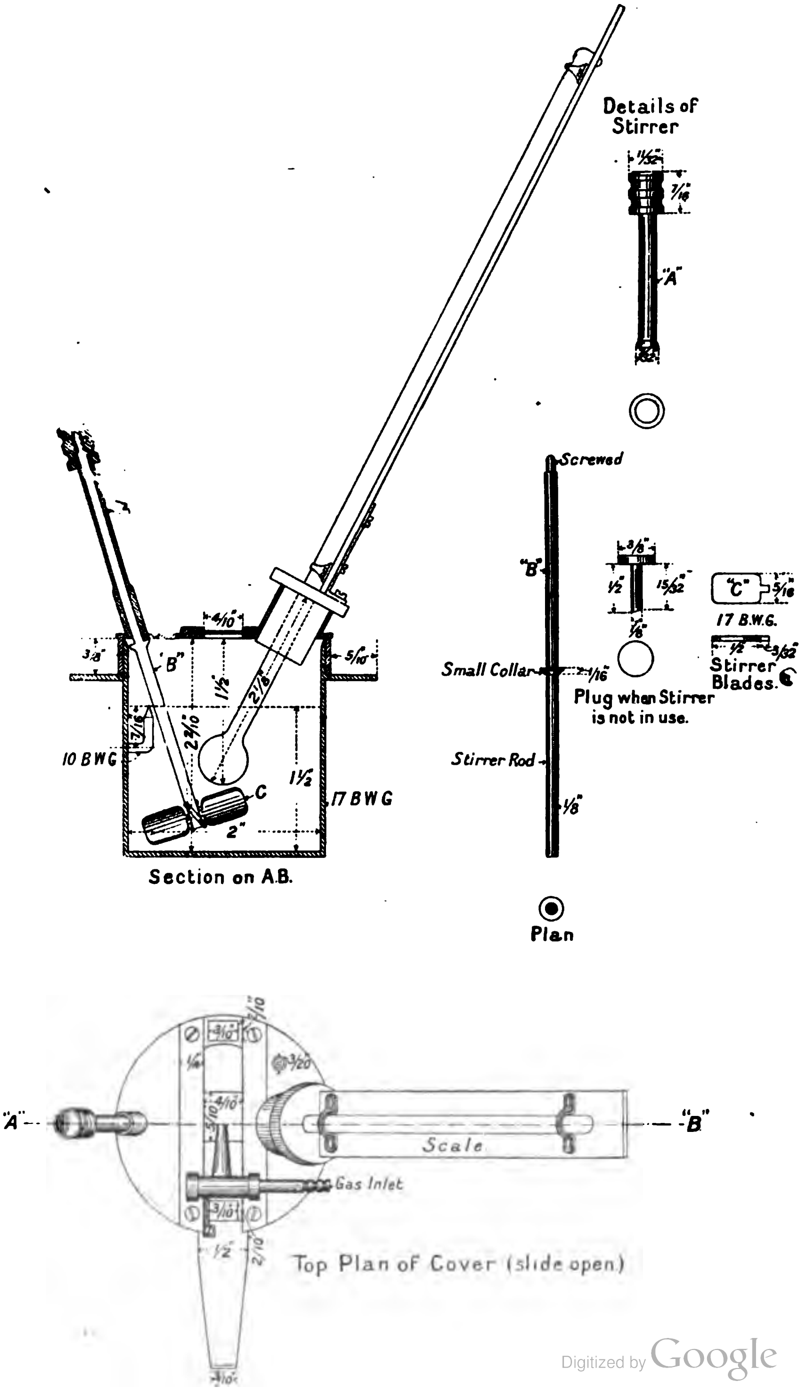

FIG. 14.—Modified Abel's cup with stirrer.

¼ inch in diameter, by means of a cork borer or other cylindrical cutter having the correct internal diameter. These cylinders are to be placed in the petroleum-cup of the testing apparatus in a vertical position in such number as will completely fill the cup. The cylinders must be in contact with one another, but must not be so tightly packed as to be deformed in shape.

"Five or six of the cylinders in the centre of the cup must be shortened to ½ inch to allow space for the thermometer bulb.

"The air-bath of the testing apparatus must be filled to a depth of 1½ inches with water. The water-bath must then be raised to and maintained at a temperature of about 75° F.

"The cup must then be placed in the air-bath, and the temperature of the sample must be allowed to rise until the thermometer in the oil-cup shows 72° F., when the test-flame must be applied.

"If no flash is obtained, this temperature must be maintained constant in the oil-cup for one hour, at the expiration of which time the test-flame must again be applied.

"If a flash is obtained, the solid mixture will be subject to the provisions of the Petroleum Acts in virtue of this Order.

"*Note.*—It may in many cases save time in testing samples of petroleum mixtures to apply the test-flame after the sample has been a few minutes in the cup and while still at the temperature of the room in which the test is being carried out, provided that this temperature is below 73° F. If a flash is obtained by this means, it is unnecessary to proceed with the test at a higher temperature."

"EFFECT OF VARIATIONS IN BAROMETRIC PRESSURE.

"It was soon found that the results obtained with the Abel instrument differed materially according to the barometric pressure at the time of testing. This source of variation appears to have been first observed accidentally in Germany, and, as the result of a series of experiments made in an air-tight chamber in the Jewish Hospital in Berlin, it was ascertained that the difference in the flashing-point amounted to about 0·30° C. for each 10 millimetres difference in the barometric column. A table of corrections was accordingly prepared in Germany for use with the Abel-Pensky tester, as described on pp. 241, 242, and 244, vol. ii.

"**Experiments at Different Altitudes in Switzerland.**—To obtain further evidence as to the effect of alteration in barometric pressure on the flashing-point, a series of experiments was conducted by the author in consultation with Sir Frederick Abel at different altitudes in Switzerland, and results exhibiting a difference of about 2° F. in the flashing-point for 1 inch difference in barometric pressure were obtained.

“ The experiments were, however, not carried out with a view of determining the exact extent of variation, the number of tests made being insufficient to form the basis of a table of corrections. The results actually obtained are given in the following table :—

“ TABLE I.—RELATION OF FLASHING-POINT TO BAROMETRIC PRESSURE.

No. of Sample.	London Bar. at 30".	Paris. 30·15".	Bouveret. 28·75".	Visp. 27·5"	Chamonix. 26·425".	Zermatt. 24·3".	Riffel. 21·5".
	° F.	° F.	° F.	° F.	° F.	° F.	° F.
1	71	71	68	66	63	60	55
2	73	73	70	68	64	62	56
3	76	76	73	71	68	65	63
4	78	78	75	73	70	67	64
5	85	85	83	80	78	75	70
Difference in barometer from London observation . .			1·25"	2·5"	3·575"	5·7"	8·5"
Mean difference in flashing-point per inch of mercurial column			2·05°	2°	2·238°	1·93°	1·76°

“ **Effect of a Tropical Climate.**—It was also found that the effect of a tropical climate upon the liberation of vapour from the oil resulted in the flashing-point being considerably lowered, and as the Indian Government, in adopting the Abel test, had copied the directions verbatim from the English Act, the result was that several cargoes of oil, which were certified as of proper flashing-point at the time of shipment, were refused admittance at the port of Calcutta.

“ **Experimental Investigations in India and in London.**—In order to investigate the matter, the author proceeded to India, and from a series of experiments made there and afterwards in England in conjunction with Sir Frederick Abel, and with the assistance of Dr Kellner, it was demonstrated that a much lower flashing-point than that furnished in a temperate climate was liable to be obtained when the Abel test was employed in a tropical climate in the manner prescribed in the English Act. Further experiments made by Dr Warden, analyst to the Government of Bengal, Professor (now Sir Alexander) Pedler, Dr Lyon, Sir Frederick Abel, Dr Kellner, and the author, showed that the depression in the flashing-point was largely due to disengagement of vapour in the act of filling the oil-cup. Although prolonged cooling of the oil minimised the liability to such disengagement of vapour, it was found that the only practical means of eliminating this source of error was to get rid of the vapour before commencing the test or before the flashing-point was reached.

"**Modifications Proposed.**—The vapour was readily removed by gently blowing over the surface of the oil before placing the cover on the cup, or by the use of an aspirator attached to the cover, or by leaving the test-slide withdrawn for some time ; but the method finally adopted consisted in commencing the test many degrees below the flashing-point, so that the vapour is withdrawn by the current of air created by the test-flame, in successive quantities too small to cause a flash before active volatilisation of the oil begins. Thus modified, the test gives results at a tropical temperature which agree very closely with those which it furnishes in a temperate climate. The slight loss of volatile constituents which occurs when the test is so performed is of no practical importance, since such loss takes place even by exposure to the air in hot climates (see a joint communication from Sir F. Abel and the author in the *Chemical News*, 2nd May 1884).

"**Directions for Conducting the Test in Temperate Climates.**—In October 1887 Sir Frederick Abel drew up the following directions for the use of the Abel instrument in *temperate* climates :—

"DIRECTIONS FOR PREPARING AND USING APPARATUS FOR TESTING PETROLEUM OIL.

"(These Directions apply to the use of the Abel tester in temperate climates.)

"(1) **Preparing the Water-Bath.**—The water-bath is filled by pouring water into the funnel until it begins to flow out at the overflow-pipe. The temperature of the water at the commencement of each test, as indicated by the long-bulb thermometer, is to be 130° F., and this is attained in the first instance by mixing hot and cold water, either in the bath or in the vessel from which the bath is filled, until the thermometer which is provided for testing the temperature of the water gives the proper indication ; or the water is heated by a spirit-lamp (which is attached to the stand of the apparatus) until the required temperature is reached.

"(2) **Preparing the Test-Lamp.**—The test-lamp is fitted with a piece of cylindrical wick of such thickness that it fills the wick-holder, but may readily be removed to and fro for the purpose of adjusting the size of the flame. In the body of the lamp, upon the wick, which is coiled within it, is placed a small tuft of cotton-wool moistened with petroleum, any oil not absorbed by the wool being removed. When the lamp has been lighted the flame is adjusted until it is the size of the bead fixed on the cover of the oil-cup.

" (3) **Filling the Oil-Cup.**—The oil-cup is placed on a level surface in a good light, and the oil to be tested is poured in without splashing, until its surface is level with the point of the gauge which is fixed in the cup. The oil should be poured from a suitable small vessel, never direct from a large can. The round-bulb thermometer is inserted into the lid of the cup, care being taken that the projecting rim of the collar touches the edge of the socket; the test-lamp, prepared as already described, is placed in position, and the cover put on to the cup and pressed down so that its edge rests on the rim of the cup.

" (4) **The Application of the Test.**—The water-bath, with its thermometer in position, is placed in some locality where it is not exposed to currents of air, and where the light is sufficiently subdued to admit of the size of the test-flame being compared with that of the bead on the cover. The cup is carefully lifted without being shaken, placed in the bath, and the test-lamp lighted. The thermometer in the oil-cup is now watched, and when the temperature has reached 66° F. (the sample, if necessary, having previously been cooled to below that temperature by immersing the vessel containing it in cold water), the operation of testing is to be commenced, the test-flame being applied for every rise of one degree.

" If the oil-cup is provided with the automatic (Pensky's) arrangement for opening and shutting the slide, the clockwork is wound up by turning the knob from left to right, and set in motion by pressing the trigger.[1]

" If the slide is intended to be moved by hand, it should be drawn open slowly and shut quickly. The exact time to be observed in this operation is regulated by the swing of the pendulum supplied with this form of instrument. The opening of the slide should take the time of three oscillations, the shutting of the slide the time of the fourth oscillation of the pendulum. (By one oscillation is meant the passage of the ball of the pendulum from the greatest distance from the vertical on the one side to the greatest distance on the other.)

" If a flash occurs at the first application of the test-flame (at 66° F.), or at any point below 73° F., the operation is to be repeated with a fresh portion of the oil, which is cooled down to 55° F. before being placed in the cup. The first application of the test-flame is made when the temperature of the oil has reached 60° F.

" In repeating a test, a fresh sample of oil must always be used, the tested sample being thrown away.

" **Correction for Atmospheric Pressure.**—As the flashing-point of an oil is influenced by changes in atmospheric pressure to an average extent of 1·6° F. for every inch of the barometer, a correction of the observed flashing-point may become necessary. The height of the barometer must, therefore, be determined at the time of making the test for the flashing-point. An aneroid barometer is supplied for this purpose. To facilitate the correction of a flashing-point for pressure a table is appended (see Table II.) giving the flashing-points of oils ranging from 65° to 80° F. (at 30 inches) under pressures ranging from 27 to 31 inches of mercury.

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[1] This refers to the Abel-Pensky apparatus, described on pp. 238, 239, 240, vol. ii., of Redwood's *Treatise on Petroleum*. (This work, pp. 32, 33, 34.)

" The table is used in the following manner :—

" *Example.*—An oil has given a flashing-point of 71° F., the barometer being 28·6 ; take the nearest number to 71° in the vertical column headed 28·6. This number is 70·8. Substitute for this number in the same horizontal line in the column headed 30 (the normal height of barometer). The substituted number—*i.e.* the true flashing-point of the oil—is 73° F.

" **Legislation in India.**—The Indian Petroleum Act, 1899, of which an abstract will be found in the section dealing with Legislation, gave power to the Governor-General in Council to alter or add to the prescribed tests. The schedule relating to testing, as modified up to 1st November 1903, is given in full in the following paragraphs. Since the Abel test was legalised in India, it has been found necessary to determine the flashing-point of Burma crude petroleum which is solid at common temperatures. In testing this oil in the Abel instrument, active evolution of vapour commences while the bulk of the oil in the cup remains solid, and the thermometer therefore does not indicate the temperature of the oil in contact with the walls of the cup from which vapour is being evolved. To meet this difficulty, the author suggested the addition of a sensitive thermometer, which is fixed in the cover of the oil-cup in a vertical position, so that the small cylindrical bulb is just immersed in the oil, and is distant only a tenth of an inch from the side of the cup. This modification has been adopted.

"TESTING-SCHEDULE OF INDIAN PETROLEUM ACT, 1899 (as modified up to 1st November 1903).

" I. NATURE OF THE TEST APPARATUS.[1]

" The apparatus consists of the following parts :—

" (1) The oil-cup.

" (2) The cover with slide, test-lamp, and clockwork arrangement for opening and closing the holes in the cover and for dipping the test-flame.

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[1] This is the Abel-Pensky apparatus hereafter described (see pp. 238, 239, 240, vol. ii., of Redwood's *Treatise on Petroleum*). (This work, pp. 32, 33, 34.)

"TABLE II.—For Correction of Flashing-Points indicated by the Test, for Variations in Barometric Pressure on either side of Thirty Inches.

Barometer in inches.																				
27	27·2	27·4	27·6	27·8	28	28·2	28·4	28·6	28·8	29	29·2	29·4	29·6	29·8	30	30·2	30·4	30·6	30·8	31
Flashing-point in degrees Fahrenheit.																				
60·2	60·5	60·8	61·2	61·5	61·8	62·1	62·4	62·8	63·1	63·4	63·7	64	64·4	64·7	65	65·3	65·6	66	66·3	66·6
61·2	61·5	61·8	62·2	62·5	62·8	63·1	63·4	63·8	64·1	64·4	64·7	65	65·4	65·7	66	66·3	66·6	67	67·3	67·6
62·2	62·5	62·8	63·2	63·5	63·8	64·1	64·4	64·8	65·1	65·4	65·7	66	66·4	66·7	67	67·3	67·6	68	68·3	68·6
63·2	63·5	63·8	64·2	64·5	64·8	65·1	65·4	65·8	66·1	66·4	66·7	67	67·4	67·7	68	68·3	68·6	69	69·3	69·6
64·2	64·5	64·8	65·2	65·5	65·8	66·1	66·4	66·8	67·1	67·4	67·7	68	68·4	68·7	69	69·3	69·6	70	70·3	70·6
65·2	65·5	65·8	66·2	66·5	66·8	67·1	67·4	67·8	68·1	68·4	68·7	69	69·4	69·7	70	70·3	70·6	71	71·3	71·6
66·2	66·5	66·8	67·2	67·5	67·8	68·1	68·4	68·8	69·1	69·4	69·7	70	70·4	70·7	71	71·3	71·6	72	72·3	72·6
67·2	67·5	67·8	68·2	68·5	68·8	69·1	69·4	69·8	70·1	70·4	70·7	71	71·4	71·7	72	72·3	72·6	73	73·3	73·6
68·2	68·5	68·8	69·2	69·5	69·8	70·1	70·4	70·8	71·1	71·4	71·7	72	72·4	72·7	73	73·3	73·6	74	74·3	74·6
69·2	69·5	69·8	70·2	70·5	70·8	71·1	71·4	71·8	72·1	72·4	72·7	73	73·4	73·7	74	74·3	74·6	75	75·3	75·6
70·2	70·5	70·8	71·2	71·5	71·8	72·1	72·4	72·8	73·1	73·4	73·7	74	74·4	74·7	75	75·3	75·6	76	76·3	76·6
71·2	71·5	71·8	72·2	72·5	72·8	73·1	73·4	73·8	74·1	74·4	74·7	75	75·4	75·7	76	76·3	76·6	77	77·3	77·6
72·2	72·5	72·8	73·2	73·5	73·8	74·1	74·4	74·8	75·1	75·4	75·7	76	76·4	76·7	77	77·3	77·6	78	78·3	78·6
73·2	73·5	73·8	74·2	74·5	74·8	75·1	75·4	75·8	76·1	76·4	76·7	77	77·4	77·7	78	78·3	78·6	79	79·3	79·6
74·2	74·5	74·8	75·2	75·5	75·8	76·1	76·4	76·8	77·1	77·4	77·7	78	78·4	78·7	79	79·3	79·6	80	80·3	80·6
75·2	75·5	75·8	76·2	76·5	76·8	77·1	77·4	77·8	78·1	78·4	78·7	79	79·4	79·7	80	80·3	80·6	81	81·3	81·6

" (3) The water-bath or heating vessel.

" (4) The tripod stand with jacket and spirit-lamp for heating the water-bath.

" (5) The thermometer for indicating the temperature of the oil in the oil-cup.

" (6) The thermometer for indicating the temperature of the water in the water-bath.

" (7) The thermometer for indicating the temperature of the oil before it is poured into the oil-cup.

" (8) The dropping bottle or pipette for replenishing the test-lamp ; and

" (9) A barometer standardised at the meteorological office of the province or at any other place appointed by the Local Government.

" The oil-cup is a cylindrical, flat-bottomed vessel made of gun-metal or brass, and tinned or silvered inside. A gauge is fixed to the inside of the cup to regulate the height to which it is to be filled with the sample under examination.

" The cup is provided with a close-fitting overlapping cover, which carries the thermometer, the test-lamp and the adjuncts thereto. The test-lamp is suspended upon two supports by means of trunnions, which allow it to be easily inclined to a particular angle and restored to its original position. The socket in the cover, which is to hold a round-bulb thermometer for indicating the temperature of the oil during the testing operation, is so adjusted that the bulb of the latter is always inserted in a definite position below the surface of the liquid.

" The cover is provided with three holes, one in the centre and two smaller ones close to the sides. These are closed and opened by means of a pivoted slide. When the slide is moved so as to uncover the holes, the suspended lamp is caught by a projection fixed on the slide, and tilted in such a way as to bring the end of the spout just below the surface of the lid. As the slide moves back so as to cover the holes, the lamp returns to its original position. Upon the cover, in front of and in a line with the nozzle of the lamp, is fixed a white bead, the diameter of which represents the size of the test-flame to be used.

" The water-bath or heating vessel is so constructed that, when the oil-cup is placed in position in it, an air-space or air-chamber intervenes between the two : consequently, in applying the test under ordinary circumstances, the heat is transmitted gradually to the oil from the hot water through the air-space. The water-bath is fitted with a socket for receiving a long-bulb thermometer, to indicate the temperature of the water. It is also provided with a funnel, an overflow-pipe, and two handles.

"The water-bath rests upon a tripod stand, which is fitted with a copper cylinder or jacket, so that the bath is surrounded by an enclosed air-space, which retains and regulates the heat. One of the legs of the stand serves as a support for a spirit-lamp, which is attached to it by a small swing bracket.

"The clockwork arrangement, by which, during the operation of testing, the slide is withdrawn, and the test-flame dipped into the cup and raised again as the slide is replaced, is provided with a ratchet key for setting it in action for each test, and with a trigger for starting it each time that the test-flame is applied.

"II. DIRECTIONS FOR DRAWING THE SAMPLE AND PREPARING IT FOR TESTING.

"(1) **Drawing the Sample.**—In all cases the testing officer or some person duly authorised by him shall personally superintend the drawing of the sample from an original unopened tin or other vessel.

"An opening sufficiently large to admit of the oil being rapidly poured or siphoned from the tin or other vessel shall be made.

"Two bottles, each of the capacity of about forty fluid ounces, are to be filled with the oil. One of these, the contents of which is intended to be preserved for reference in case of need, is to be carefully corked, the cork being well driven home, cut off level with the neck, and melted sealing-wax worked into it. The other bottle may be either stoppered or corked.

"(2) **Preparing the Sample for Testing.**—About ten fluid ounces of the oil, sufficient for three tests, are transferred from the bottle into which the sample has been drawn to a pint-flask or bottle, which is to be immersed in water artificially cooled until a thermometer, introduced into the oil, indicates a temperature not exceeding 50° F.

"III. DIRECTIONS FOR PREPARING AND USING THE TEST APPARATUS.

"(1) **Preparing the Water-Bath.**—The water-bath is filled by pouring water into the funnel until it begins to flow out at the overflow-pipe. The temperature of the water at the commencement of each test, as indicated by the long-bulb thermometer, is to be 130° F., and this is attained in the first instance by mixing hot and cold water, either in the bath or in a vessel from which the bath is filled, until the thermometer which is provided for testing the temperature of the water gives the proper indication; or the water is heated by means of the spirit-lamp (which is attached to the stand of the apparatus) until the required temperature is indicated.

"(2) **Preparing the Test-Lamp.**—The test-lamp is fitted with a piece of cylindrical wick of such thickness that it fills the wick-holder, but may readily be moved to and fro for the purpose of adjusting the size of the flame. In the body of the lamp, upon the wick, which is coiled within it, is placed a small tuft of cotton-wool, moistened with petroleum, any oil not absorbed by the wool being removed. When the lamp has been lighted the wick is adjusted by means of a pair of forceps until the flame is of the size of the bead fixed on the cover of the oil-cup; should a particular test occupy so long a time that the flame begins to get smaller, through the supply of oil in the lamp becoming exhausted, three or four drops of petroleum are allowed to fall upon the tuft of wool in the lamp from the dropping bottle or pipette provided for that purpose. This can be safely done without interrupting the test.

"(3) **Filling the Oil-Cup.**—The oil-cup having been previously cooled, by placing it bottom downwards in water at a temperature not exceeding 50° F., is to be rapidly wiped dry, placed on a level surface in a good light, and the oil to be tested is poured in very slowly, without splashing, until its surface is level with the point of the gauge which is fixed in the cup. The round-bulb thermometer is inserted into the lid of the cup, care being taken that the projecting rim of the collar touches the edge of the socket; the test-lamp, prepared as already described, is placed in position, and the cover is then put on to the cup and pressed down so that its edge rests on the rim of the cup.

"(4) **Application of the Test.**—The water-bath, with its thermometer in position, is placed in some locality where it is not exposed to currents of air, and where the light is sufficiently subdued to admit of the size of the entire test-flame being compared with that of the bead on the cover. The cup is carefully lifted without shaking it, and placed in the bath, the test-lamp is lighted, and the clockwork wound up by turning the key. The thermometer in the oil-cup is now watched, and when the temperature has reached 56° F., the clockwork is set in motion by pressing the trigger.

"If no flash takes place, the clockwork is at once re-wound, and the trigger pressed at 57° F., and so on, at every degree of rise of temperature, until the flash occurs, or until a temperature of 95° F. has been reached.

"If the flash takes place at any temperature below 77° F., the temperature at which it occurs is to be recorded. Two fresh portions of the sample are then to be successively tested in a similar manner, and the results recorded. If no greater difference than 2° F. exists between any two of the three recorded results, and if in no instance the flash has taken place within eight degrees of the temperature at which the testing is commenced, each result is to be corrected for atmospheric pressure as hereafter described, and the average of the three corrected results is the flashing-point of the sample. In the event of there being a greater difference than 2° F. between any two of the results, while in no instance has the flash taken place within eight degrees of the temperature at which the testing was commenced, the series of tests is to be rejected, and a fresh series of three similarly obtained, and so on, until a sufficiently concordant series is furnished, when the results are to be corrected, and the average taken in the manner already described.

"If, however, a flash has occurred at or below 64° when the test is applied in the manner above described, the next testing shall be commenced ten degrees lower than the temperature at which the flash had been previously obtained (that is to say, at 54° or thereunder), and this procedure shall be continued until the results of three consecutive tests do not show a greater difference than two degrees and until a flash has not occurred in any of the three tests within eight degrees of the temperature at which the testing is commenced: Provided always that, if at the commencement of the series of tests a flash has occurred on the first application of the test-flame at 56°, and if a flash has also occurred on the first application of the flame in each of three successive tests in which, thereupon, the test-flame is first applied at 46° as above directed, the testing officer shall certify that the petroleum has a flashing-point below 47°, and the sample shall be reported dangerous.

"If a temperature of 76° F. has been reached without a flash occurring, the application of the test-flame is to be continued at every degree of rise of temperature until a temperature of 95° F. has been reached. If no flash has occurred up to this point, and if the petroleum is declared to be imported subject to the provisions of the Act, the tests shall not be continued, and the testing officer shall certify that the petroleum has a flashing-point over 95° F. and is not dangerous. But, if the petroleum is oil ordinarily used for lubricating purposes, and is declared to have its flashing-point at or above 200°, or is oil to which a notification of the Local Government exempting it from the operation of the Act will be applicable in the event of the flashing-point being found to be at or above 120°, the test shall be continued as follows:—The oil-cup is to be removed from the water-bath, and the temperature of the water in the water-bath is to be reduced to 95° F. by pouring cold water into the funnel (the hot water escaping by the overflow-pipe). The air-chamber is then to be filled to a depth of 1½ inches with water at a temperature of about 95° F., the oil-cup is to be replaced in the water-bath and the spirit-lamp attached to the water-bath is to be lighted and placed underneath. The test-flame is then to be again applied from 96° F., at every degree rise of temperature as indicated by the thermometer in the oil-cup until a flash takes place, or until a temperature of 200° F. or 120° F., as the case may be, has been reached. If during this operation the test-flame appears to diminish in size, the lamp is to be replenished in the manner prescribed at 2 without interrupting the test.

"If a flash occurs at any temperature between 76° and 200° F., the temperature at which it occurs, subject to correction for atmospheric pressure, is the flashing-point of the sample.

"In repeating a test a fresh sample of oil must always be used, the tested sample being thrown away, and the cup must be wiped dry from any adhering oil and cooled, as already described, before receiving the fresh sample.

"(5) **Correction for Atmospheric Pressure.**—As the flashing-point of an oil is influenced by changes in atmospheric pressure to an average extent of 1·6° F. for every inch of the barometer, a correction of the observed flashing-point may become necessary. The height of the barometer must therefore be determined at the time of making the test for the flashing-point. The true height of the barometer for the purpose of the test shall be considered to be the height of the column of mercury measured at 32° F., which is supported by the air pressure at the time of the experiment; that is, the actual height of the barometer at the time of observation duly corrected for any error of the instrument and for its temperature if necessary. For the purpose of applying the correction to the flashing-point of the oil obtained by the test, a table (see Table II.) is appended to this schedule giving the flashing-points of oils ranging from 65° to 80° F. under pressures ranging from 27 to 31 inches of mercury.

"The table is used in the following manner:—

"*Example.*—An oil has given a flashing-point of 71°, the barometer being at 28·6 inches; take the nearest number to 71° in the vertical column headed 28·6. This number is 70·8°. Substitute for this the number in the same horizontal line in the column headed 30 (the normal height of the barometer). The substituted number, that is, the true flashing-point of the oil, is 73°.[1]

"(6) **Application of the Test to Viscous Fluids or Preparations, such as Solutions of Indiarubber in Mineral Naphtha, or Thick Paint made with that Material.**—About a teaspoonful of the substance to be tested is placed in the cup, and the cover fitted with a thermometer is put on.

"The cup thus prepared for the test is then cooled down until the thermometer indicates a temperature of 50° F. This may be accomplished either by placing the prepared cup in a refrigerator, or by immersing it up to its projecting collar in water, which is maintained at a sufficiently low temperature until the result specified has been obtained.

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[1] This paragraph was added by Notification No. 928 (J), dated 28th June 1900. See *Gazette of India*, 1900, Part I. p. 403.

"The prepared cup thus cooled is then transferred to the water-bath, the temperature of which has previously been raised to 70° F. (The scale of the thermometer in the water-bath should range from 60° to 180° F.)

"The test is then applied as described in section 4 of this part. If no flash has taken place when the temperature in the cup has reached 75° F., the test need not be continued.

"The temperature at which the flash occurs is the observed flashing-point of the substance, and, subject to correction for atmospheric pressure as prescribed in the Act, is the true flashing-point.

"IV. DIRECTIONS FOR DETERMINING THE FLASHING-POINT OF PETROLEUM WHICH IS NOT FLUID AT ORDINARY TEMPERATURES.

"(1) **Nature of the Test Apparatus.**—The instrument employed is the Abel-Pensky petroleum testing apparatus, fitted with an additional thermometer to indicate the temperature of the oil in close proximity to the walls of the cup. This thermometer has a cylindrical bulb, $\frac{7}{8}$ inch in length and $\frac{3}{16}$ inch in diameter. It is scaled from 45° to 165° F., ten degrees on the scale occupying $\frac{3}{8}$ inch. The thermometer is held vertically in a socket attached to the cover of the oil-cup in such a position that the bulb is $\frac{1}{16}$ inch from the side of the cup.

"(The thermometer can be removed and the orifice which is provided for it closed by means of an indiarubber plug, if the apparatus is required for testing petroleum in the ordinary way.)

"(2) **Directions for Preparing the Sample for Testing.**—About ten fluid ounces of the oil are placed in a pint-flask, the mouth of which is then closed with an indiarubber stopper and the sample is liquefied by placing the flask in a water-bath, the temperature of which is only raised sufficiently high to liquefy the oil.

"(3) **Directions for Preparing and Using the Test Apparatus.**—The water-bath and test-lamp are to be prepared in the manner prescribed in Part III. of this schedule. The oil-cup is to be filled with the liquefied oil, and the cover (into which both thermometers are to be previously inserted) placed on it, care being taken that the bulb of the additional thermometer is not brought into contact with the bracket-gauge fixed inside the cup. The oil-cup is then to be placed in a refrigerator, or plunged up to the projecting collar in water maintained at sufficiently low temperature, until both thermometers indicate the temperature at which the testing of petroleum is directed in Part III. of this schedule to be commenced. The oil-cup is then to be removed, wiped dry and placed in the water-bath, and the testing effected in the manner prescribed in Part III. of this schedule, the temperature indicated by the additional (vertical) thermometer alone being noted, and the average of three determinations, duly corrected for atmospheric pressure, being recorded as the flashing-point of the sample, provided that no greater difference than 4° F. exists between any two of such results."

"**Use of the Abel Test in Other Countries.**—The various countries where the Abel tester in its original and modified forms are in use, and the respective minimum flashing-points prescribed, are given under Legislation.

"**The Abel-Pensky Instrument.**—In 1880 the subject of petroleum-testing was investigated in Germany on behalf of the Government, and the Abel instrument was selected as the best, but, as exception was taken to the personal error considered to be liable to be introduced by the method of applying the test-flame, the modification devised by Pensky, of Berlin, was adopted. The modification consists in effecting the movement of the slide and application of the test-flame by a clockwork arrangement, which ensures uniformity. A section of the instrument—which in all other respects is practically identical with the ordinary Abel apparatus—and the details of the clockwork mechanism are shown in figs. 15 and 16, which also give the dimensions in millimetres.

"The slide, S, pivoted on the centre *z*, is set in motion by the arm *d*, moving on a central axis. This arm carries on its under side two pins, e_1, e_2. When the slide is in its normal position—*i.e.* closing the openings, o_1, o_2, o_3,—one of the pins, e_1, rests against the steel plate *f*, on the right-hand side of the slide, and the other, e_2, is held by the catch *g*. On the axis of the arm *d* is fastened the spring-case *u* containing a spiral spring.

"To wind up the clockwork, the knob *b* is turned half round, further winding being prevented by a stop. To move the slide, the trigger *k* is pushed towards the spring *h*, the catch *g* being thus drawn back, and the pin e_2 released. The spiral spring unwinds and presses the other pin, e_1, against the slide S, moving this slide aside to the left, and uncovering the apertures. As soon as the pin e_1 is carried by the revolution of the arm *d* past the steel plate *f*, the slide is forced back to its former position by the spring *r*, and the pin e_1 is stopped by the catch *g*, which returns it to its original place as the trigger *k* is released. These movements are repeated every time the test is applied. The uncoiling of the spiral spring and movement of the slide are regulated by the escapement *m n p*. The three holes in the lid of the oil-cup, and the two holes in the slide (the lateral movement of the slide uncovering and covering the third hole in the lid) are of the same dimensions as those in the Abel instrument, but not quite the same shape, on account of the circular motion of the slide. The lamp is tipped by a nosepiece *n*, and on its return to the perpendicular, is prevented from swinging backwards by a stop *v*.

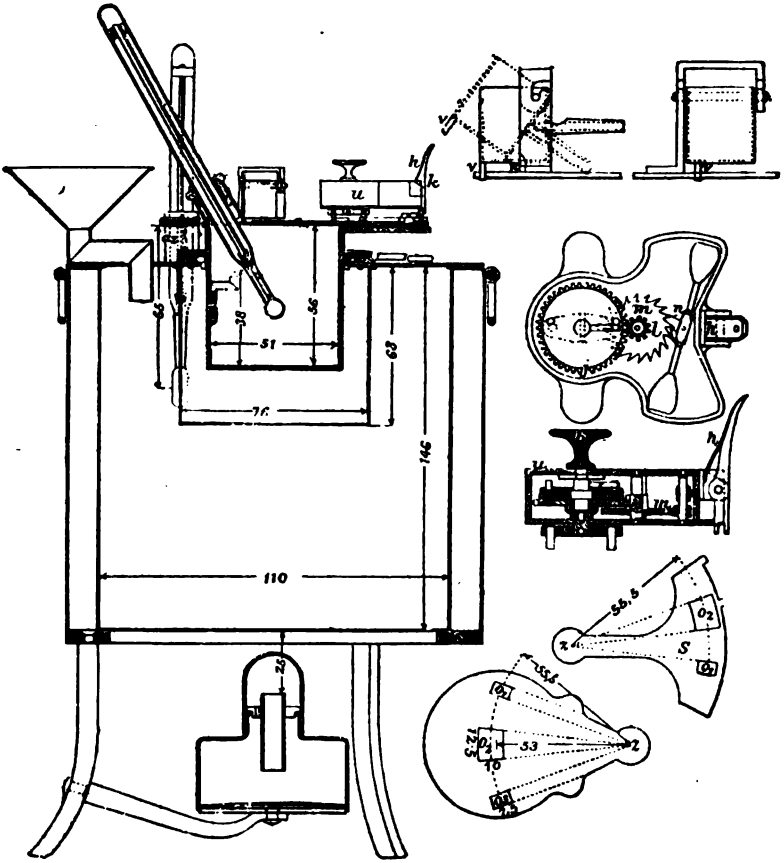

Fig. 15.—Abel-Pensky testing instrument.

"**Directions for the Use of the Instrument.**—The following is the method of applying the Abel-Pensky test prescribed in Germany[1]:—

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[1] *Vorschriften betreffend den Abelschen Petroleumprober und seine Anwendung*, Berlin, 1883.

"The testing should be carried out in an apartment of medium temperature and free from draughts. The petroleum samples should be in closed vessels and have been long enough in the room to have acquired the same temperature, otherwise the tests will not be satisfactory.

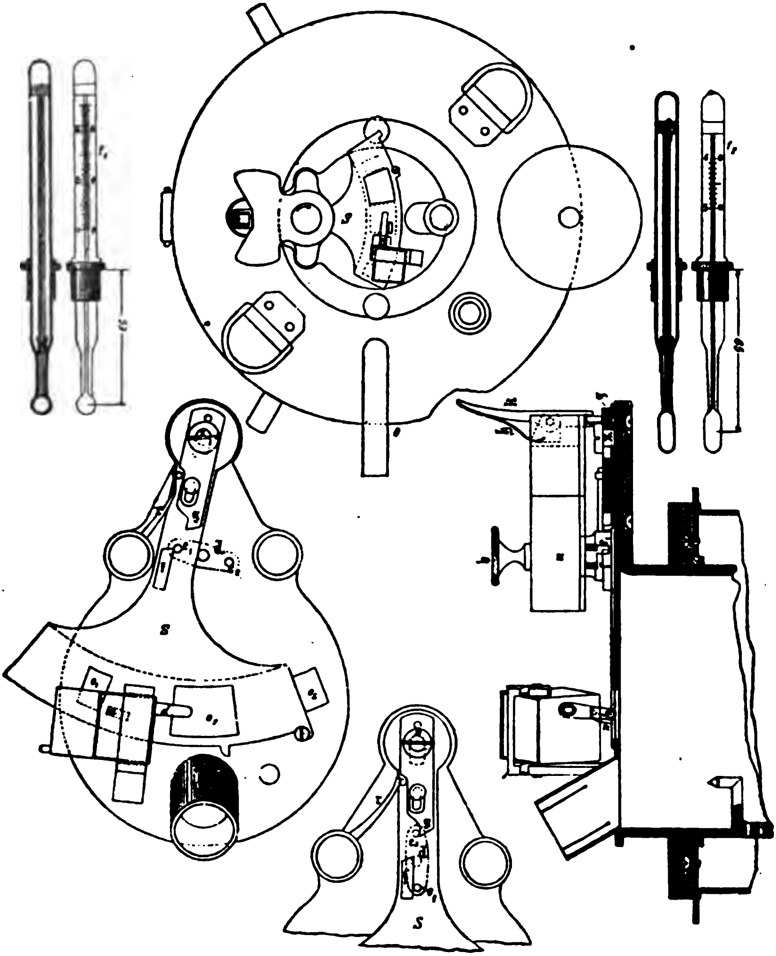

FIG. 16.—Abel-Pensky testing instrument.

"The height of the barometer must be observed before commencing operations, and the temperature at which the test is to be first applied varies with the pressure indicated, according to the following table :—

"TABLE III.—INITIAL TEST-POINT FOR VARIOUS BAROMETRIC PRESSURES.

Height of Barometer.	First Test applied at
685 to 695 millimetres	+14° C.
695 ,, 705 ,,	+14° C.
705 ,, 715 ,,	+14·5° C.
715 ,, 725 ,,	+15° C.
725 ,, 735 ,,	+15·5° C.
735 ,, 745 ,,	+16° C.
745 ,, 755 ,,	+16·5° C.
755 ,, 765 ,,	+17° C.
765 ,, 775 ,,	+17° C.
775 ,, 785 ,,	+17·5° C.

If the barometer varies from the normal pressure of 760 mm., by more than 2½ mm. either way, the actual flashing-point observed is corrected in accordance with Table IV. The table being arranged for intervals of 5 mm., intermediate pressures are counted as 0 or 5, according to the figure which they most nearly approach—viz. 742 counts as 740, 743 as 745, and so on. The instrument must be set level on the testing-table, with the aid of a spirit-level or plumb-line, before it is filled, and it should be at such a height that the red mark on the water-bath thermometer is on a level with the eye of the operator. The bath is next filled with water at +50° to 52° C. until a portion runs out through the overflow-pipe, to which an indiarubber tube should be attached to convey the surplus water away without splashing the apparatus or table. If cold water is used for filling the bath, it must be warmed by the heating lamp before putting in the oil-cup, care being taken that the base ring does not get overheated. The test-lamp is prepared for use by being packed with loose cotton-wool, which is then saturated with petroleum, any surplus being poured out. If this is not attended to, the tipping of the lamp at the moment of applying the test might cause drops of oil to fall on to the lid of the tester and vitiate the results. The wick must also be cleansed from any adherent incrustation or char. The oil-cup, with its cover and thermometer, are to be thoroughly clean and dry, and all traces of oil from preceding tests are to be removed by drying and absorption. Finally, the petroleum to be tested, if not at least two degrees cooler than the lowest of the temperatures recorded in the foregoing table, must be cooled down to that point, together with the oil-cup, the latter being dipped into cold water. To obtain very exact results, it is advisable to use water cooled to +11° C. for this purpose, and to keep the oil-cup in it long enough for proper cooling. Before inserting the oil-cup, the water-bath is heated up to 54° to 55° C., the temperature indicated by the red mark on the thermometer-scale. Meanwhile, the oil is poured into the oil-cup by means of the pipette, until the extreme point of the indicator just projects above the surface of the liquid. It is important that this limit should not be exceeded, but if this should happen, the cup must be emptied, dried carefully, and refilled. Any bubbles forming on the surface of the oil must be dissipated by pricking with the warm charred point of a burned-out match. The table on which the cup stands during the operation of filling should be perfectly horizontal, and quite near to the water-bath, to minimise the risk of tilting or shaking the cup. The oil is poured into the centre of the cup, and not against the sides, and to prevent bubbles, the nozzle of the pipette should be kept below the surface of the liquid. The utmost exactness is obtained by slowly adding the oil by a pipette until the point of the indicator is just about to disappear under the surface of the liquid, and then removing the excess by the insertion of a small pipette. Bubbles formed by emptying the cup may be removed on refilling, by a warm metallic point or a charred match. The lid of the cup is put on directly after pouring in the oil, the thermometer being previously inserted in its socket, and pressed down tightly, avoiding contact between the ebonite plate under the clockwork and the knobs, K, on the bath, as this would prevent the lid from fitting properly. The insertion of the oil-cup into the bath must be done without tilting or shaking, and to avoid the risk of this, the cup, previously cooled by ice and water to 8° C. (to prevent the oil getting warm while filling), may be put into position before filling. Should the water-bath be at a higher temperature than 54° to 55° C., it must be cooled by the addition of cold water, and when that degree is reached, the lamp is extinguished.

"When the oil approaches the temperature at which the first test is to be applied, in accordance with the foregoing table, the test-lamp is lighted and the flame adjusted until, when viewed from the front, it is of the same width as the white bead on the lid. This is important, as the size of the flame has an influence on the flashing-temperature. The clockwork is then wound up by turning the milled head as far as it will go in the direction indicated by the arrow. On releasing the catch by pressing on the trigger, the slide slowly and regularly moves aside, and at the end of two seconds has returned to its original position. During the movement of the slide, the behaviour of the test-flame as it approaches the surface of the oil must be carefully observed, particular attention being directed to sheltering the apparatus from all draughts and from the breath of the operator. This is facilitated by the provision of a glass plate fixed to the draw-out side of the case containing the apparatus. The test is to be repeated at every ½° rise of the thermometer until a flash is obtained. Before the absolute flashing-point is reached, the size of the test-flame will be observed to increase by a kind of halo when dipped towards the oil, but the true flash is a bluish lightning-like flame extending over the whole free surface of the oil. The temperature indicated by the thermometer is subject to correction, both as regards barometric pressure and error, if any, of the thermometer itself, according to the variation ascertained on standardising the instrument. The test is repeated with a fresh portion of the same oil, after cooling the oil-cup and lid, drying and freeing them, as well as the thermometer, from all adherent drops of oil, and reheating the bath to 55° C.

"If the result of the second test agrees with the first within ½°, the average is taken as the apparent flashing-point. If, however, the difference is 1° or more, a third test is necessary; and provided the results of the three do not differ more than 1½°, the average is taken. In case of a greater divergence, a fresh set of tests must be made. In the case of oils flashing at the first application of the test-flame the actual flashing-point may be higher than is indicated by the thermometer, the accumulation of vapour in the oil-cup causing the flash to occur sooner than it would under ordinary circumstances. If it is desired to test such oils accurately, the operation is begun at a lower temperature than usual, and repeated at lower degrees until no flash is produced on first opening the slide.

"The apparent flashing-points must be corrected for pressure, according to Table IV., by finding the observed flashing-point in the column headed by the existing height of the barometer, and proceeding in the same row to the column headed 760, which will show the actual flashing-point at normal pressure. Fractions between the figures given in the table are to be counted as the figure to which they most nearly approach. Any error recorded by the standardiser of the apparatus must be corrected before applying the correction in the table.

"**The Standardising of the Abel-Pensky Instrument.**—By an order of the Imperial Chancellor, dated 21st July 1882, the Kaiserliche Normal-Aichungs-Kommission was empowered to standardise and stamp instruments for testing petroleum, particular attention being devoted to the following points:—

"(*a*) The dimensions of the apparatus.

"(*b*) The accurate timing of the slide movement.

"(*c*) The accuracy of the thermometer.

"(*d*) The correctness of the flashing-points indicated by the apparatus.

“TABLE IV.—Correction of Apparent Flashing-Points to the Normal Barometric Pressure of 760 Millimetres.

Millimetre pressure.																				
685	690	695	700	705	710	715	720	725	730	735	740	745	750	755	760	765	770	775	780	785
Flashing-points, Centigrade scale.																				
16·4	16·6	16·7	16·9	17·1	17·3	17·4	17·6	17·8	18·0	18·1	18·3	18·5	18·7	18·8	19·0	19·2	19·4	19·5	19·7	19·9
16·9	17·1	17·2	17·4	17·6	17·8	17·9	18·1	18·3	18·5	18·6	18·8	19·0	19·2	19·3	19·5	19·7	19·9	20·0	20·2	20·4
17·4	17·6	17·7	17·9	18·1	18·3	18·4	18·6	18·8	19·0	19·1	19·3	19·5	19·7	19·8	20·0	20·2	20·4	20·5	20·7	20·9
17·9	18·1	18·2	18·4	18·6	18·8	18·9	19·1	19·3	19·5	19·6	19·8	20·0	20·2	20·3	20·5	20·7	20·9	21·0	21·2	21·4
18·4	18·6	18·7	18·9	19·1	19·3	19·4	19·6	19·8	20·0	20·1	20·3	20·5	20·7	20·8	21·0	21·2	21·4	21·5	21·7	21·9
18·9	19·1	19·2	19·4	19·6	19·8	19·9	20·1	20·3	20·5	20·6	20·8	21·0	21·2	21·3	21·5	21·7	21·9	22·0	22·2	22·4
19·4	19·6	19·7	19·9	20·1	20·3	20·4	20·6	20·8	21·0	21·1	21·3	21·5	21·7	21·8	22·0	22·2	22·4	22·5	22·7	22·9
19·9	20·1	20·2	20·4	20·6	20·8	20·9	21·1	21·3	21·5	21·6	21·8	22·0	22·2	22·3	22·5	22·7	22·9	23·0	23·2	23·4
20·4	20·6	20·7	20·9	21·1	21·3	21·4	21·6	21·8	22·0	22·1	22·3	22·5	22·7	22·8	23·0	23·2	23·4	23·5	23·7	23·9
20·9	21·1	21·2	21·4	21·6	21·8	21·9	22·1	22·3	22·5	22·6	22·8	23·0	23·2	23·3	23·5	23·7	23·9	24·0	24·2	24·4
21·4	21·6	21·7	21·9	22·1	22·3	22·4	22·6	22·8	23·0	23·1	23·3	23·5	23·7	23·8	24·0	24·2	24·4	24·5	24·7	24·9
21·9	22·1	22·2	22·4	22·6	22·8	22·9	23·1	23·3	23·5	23·6	23·8	24·0	24·2	24·3	24·5	24·7	24·9	25·0	25·2	25·4
22·4	22·6	22·7	22·9	23·1	23·3	23·4	23·6	23·8	24·0	24·1	24·3	24·5	24·7	24·8	25·0	25·2	25·4	25·5	25·7	25·9

"(*e*) The accuracy of the metallic barometer supplied with the instrument.

"The examination of the thermometers should not take place until they have been delivered.

"In verifying the dimensions of the apparatus, the following variations from the exact standard are allowed in :—

	Millimetres.
"The thickness of the metal plates,	0·2
"The diameter of the lamp-spout,	0·2
"The distance of the point of the filling-indicator from the upper edge of the cup,	0·5
"The distance from the lower corner of the inside of the lamp-spout to the upper face of the cup-lid when the lamp is fully tipped,	0·5
"The thickness of the ebonite ring,	0·5
"The inside diameter and height of the oil-cup ; the width of the brass ring ; its distance from the upper edge of the cup ; the distance of the axis of the slide from the centre of the cup-lid and the centre of the three apertures ; the diameter of the bead ; and the dimensions of the tube for the insertion of the thermometer, t_1,	1·0
"The distance of the centre of the bulb of the thermometer, t_1, from the end of the sheathing tube and from the underside of the cup-lid,	1·0
"The dimensions of the water-bath, and of the outer cylinder ; the width of the ebonite ring on the bath ; and the distance of the lowest mark on the scale of either thermometer from the enlargement on the tube,	2·0

"The variation of time allowed for the slide movement is 0·2 second over or under the prescribed standard. The thermometer, t_1, may not vary from the normal instrument more than 0·2° C. when tested in four places on the scale, and the thermometer, t_2, not more than 0·5° C. when tested in two places. Furthermore, successive portions of the same sample of oil, consecutively examined, should not show a greater divergence of flashing-point than 0·75° C., and the average of five or seven such tests should not differ from the average of the same recorded by the standard by more than 0·5° C., an oil approximating to the standard flashing-point being used for the experiment. The metallic barometer belonging to the apparatus should not differ more than 2 mm. from the standard at the ordinary room temperature. It must be examined with a view to ascertaining the influence of sudden alterations of pressure or continued shaking of the instrument on its accuracy between the limits of 680 and 790 mm. The instruments which satisfy the tests imposed are stamped by the Normal-Aichungs-Kommission on all the removable portions, and the amount of variation or error of flashing-point to be allowed for is indicated. The maker's name, the number of the instrument, and the year of examination are included in the stamping, and all the particulars are inscribed on a certificate issued by the standardiser, together with the following measurements :—

"(*a*) Distance of the point of the filling-indicator from the upper edge of the oil-cup.

"(*b*) Distance of the lowest inside point of the lamp-spout from the under surface of the cup-lid when the lamp is fully tilted.

"(*c*) Distance of the central point of the oil-thermometer bulb from the under surface of the cup-lid.

"To guard against any alteration on these points subsequent to standardising, the Normal-Aichungs-Kommission send out with each apparatus examined, a stamped gauge consisting of a rectangular steel plate, on one side of which is a projection for controlling the measurement, *a* ; on the other a second projection bounded by two flutings, to measure, *b*, and furnished with a projecting stud on one of its flat surfaces for gauging, *c*. To examine *a*, the gauge is applied to the edge of the oil-cup so that the projection hangs down inside and the mark on it is touched by the point of the filling-mark. For the second verification, *b*, the clockwork is set in motion, and the apertures in the lid are wedged open so that the mouth of the lamp is at its lowest point. The gauge is then applied under the lid, and the lower inside corner of the spout should coincide with the end of the smaller projection. For examining *c*, the side of the gauge opposite to the smaller projection used for *b* is applied to the under side of the lid, when the stud should exactly touch the middle of the bulb of the thermometer. The length of the lamp-spout can also be verified, a mark cut at right angles to the plain side of the gauge showing the exact size. The edge of the spout-mouth should coincide with the mark ; and should either the top or bottom corner fail to do so, the apparatus is out of adjustment.

"In 1907 an International Commission for the Unification of the Methods of Testing Petroleum Products[1] was appointed, with Dr Leo Ubbelohde, of the Technical High School, Karlsruhe, Baden, as General Secretary. It had long been known that the German Abel-Pensky instrument gave results at least 3° F. lower than those furnished by the English Abel apparatus, and the Commission commenced its labours by undertaking an examination and comparison of various

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[1] Now called the International Petroleum Commission.

methods of determining the flash-point of petroleum. At the invitation of the English section of the Commission, the National Physical Laboratory undertook to assist in the investigation, and the results reached in the first stage of this work have been given in a report by Dr J. A. Harker, F.R.S., senior assistant in the Physics Department, and Mr W. F. Higgins, Junior Assistant in the Physics Department. The report, which was issued during 1911, gives full details of the experimental investigation carried out, and is divided under the following heads :—On the apparatus and methods used in petroleum testing ; Thermometry ; Accuracy attainable in flash-point determinations ; The general theory of flash-point determination ; Temperature distribution ; The heating effect of the test-flame ; Comparisons of the flash-points obtained on different types of apparatus ; Summary and general results of the investigation ; Appendix, Section 1, flash-points of mixtures of oils ; Appendix, Section 2, notes to the tables of dimensions ; Appendix, Section 3, the 'lag' of the different types of thermometers employed. The instruments employed were the English Abel apparatus, the British Colonial type of Abel-Pensky apparatus, and the German Abel-Pensky apparatus. The average values of the differences between the flash-point as obtained in the Abel apparatus and on the other types were found to be :—

"Colonial type of Abel-Pensky, 1·1° F. higher.
"German type of Abel-Pensky, 3·7° F. higher.

"The details of the experiments made afford an explanation of the causes of these differences, as is indicated in the following summary :—

"The main result of the investigation thus far has been to ascertain what is almost certainly the true cause of the differences in the flash-points given by specimens of the same type of apparatus and by those of different types. The results may be summarised as follows :—

"(1) The influence of variations in the method of procedure in making a test has been studied ; the points investigated include the effect of :

"(*a*) The frequency of application of the test-flame ;
"(*b*) Variations in the time of opening of the slide ;
"(*c*) Variations in the water-bath temperature ;
"(*d*) Variations in the depth of immersion of the thermometer.

"(2) The temperature distribution in the oil-cup was investigated, and it was found that large systematic differences of temperature between different parts of the apparatus were always present ; also that the temperature distribution was not the same in the different types of apparatus. This has been investigated in full for the Abel and Colonial type of Abel-Pensky apparatus ; and also to some extent in the case of the German Abel-Pensky. The differences found between the three forms of apparatus are satisfactorily and completely explained by the results of these experiments on temperature distribution.

"(3) It was found that the heat from the test-flame was a factor of considerable importance in the carrying out of a flash-point test, and that, owing to the difficulty of always setting the flame to the same size, differences of $\pm\frac{1}{2}$° F. are to be expected even in the most careful work, more especially in the apparatus provided with the oil test-flame.

"(4) The difference between the flash-points obtained with the different types of tester has been investigated and has been found to be approximately constant at all temperatures. It might be noted here that a difference has been found to exist between the results obtained with the two forms of Abel apparatus, fitted with oil and gas test-flames respectively; the latter reads lower by about $\frac{1}{2}$° F. Further systematic comparisons between all the different types of apparatus are contemplated.

"(5) It must be recognised that the flash-point of a liquid, as ordinarily determined, is an empirical constant, which is largely a function of the particular apparatus used. It would seem desirable, therefore, to establish, if possible, a more definite connection between this property and the real physical properties of the substance. Experiments are now in progress to study the relation subsisting between 'flash-point' and vapour pressure, both with oils and other substances, and it is hoped that out of these experiments it may be possible to establish conditions under which certain definite liquids may be used for the standardisation of different forms of flash-point apparatus."

The Gray Tester.—The development of this instrument, now so largely used by petroleum chemists, is explained by the following extract from (*a*) the report of a meeting of Scottish Mineral Oil Works Chemists held in connection with the Society of Chemical Industry, and recorded in the *Journal* of that Society for 1891, and also by the paper (*b*) read on the above occasion by Mr John Gray, F.I.C., entitled "An Apparatus for Determining the Flash-Points of Heavy Mineral Oils."

As already indicated on p. 6, the apparatus is illustrated by figs. 11 and 12.

(*a*) "FLASHING-POINTS OF HEAVY MINERAL OILS.

"Determination of the flashing-point of heavy mineral oils.

"The use of the covered oil-cup of the 'Abel' or Government flash-point apparatus for burning oils has gradually become general for the purpose of determining the flash-point of heavy or lubricating oils as well. Its use for this purpose is, however, attended with one rather serious drawback, which is, that when used for high temperatures there is no arrangement for regulating the rate at which the oil is heated. When the Abel's cup is used for heavy oils, it is usually heated by means of a small naked flame or on a sand-bath, and with a view of making the rate of heating as regular and uniform as possible, the committee specified that not less than 15 minutes should be taken in raising the temperature of the oil to 300° F.; when this is done fairly concordant results are obtainable.

"When the committee first met, Mr Calderwood brought under their notice a German apparatus, designed by Pensky and Marten, which he reported as yielding most satisfactory results, both in Mr Redwood's hands and his own. A short description of this apparatus is to be found in this *Journal*, viii. 734, and the principal feature which distinguishes it from the Abel arrangement is, that it is provided with a stirrer, which ensures not only a thorough agitation of the oil, but also of the air or vapour above the oil.

"Experiments showed that the discrepancies which frequently occurred with the old method arose from local overheating of the oil as much as anything else, and that when a stirring arrangement is attached to the oil-cup to prevent this, the rate of heating within wide limits was practically without effect upon the result. In these circumstances it was, therefore, specified that in the event of a dispute arising as to the correct flashing-point of a heavy mineral oil, it was to be decided by means of the Pensky-Marten apparatus.

(*b*) "AN APPARATUS FOR DETERMINING THE FLASH-POINTS OF HEAVY MINERAL OILS.[1]

"The necessity for a speedy and accurate method of determining the flash-points of heavy mineral oils is fully recognised by those who are called upon to make numerous tests, and perhaps no one is more alive to the uncertainty of the methods which are at present employed than the chemists of the Scotch mineral oil works.

"It is hardly necessary for me to refer to the old method of taking flash-points, known as the 'open test,' which is well known to give discordant results, even in the hands of the same operator, according to variable and almost uncontrollable conditions under which the test may be carried out. It is still recommended as 'sufficiently accurate for many practical purposes.'

"The above method, though still employed in some works laboratories, is gradually being superseded by the Abel's cup test, in which the oil-cup and cover of the well-known petroleum tester are employed. As already mentioned by Mr Thompson, this method has been adopted by the committee of oil works chemists, provided the time in heating to 300° F. be not less than 15 minutes. I have found that the flash-point of an ordinary Scotch 885° oil will vary as much as 6° F. according as the time of heating varies between 15 and 90 minutes, when the test is carried out with the ordinary Abel's cup. It would therefore be necessary to state the maximum as well as the minimum time of heating, in order to make the results indisputable. With oils having such a wide range of flash-points as those we are dealing with, the fixing of an absolute time is a practical impossibility.

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[1] The apparatus is made by, and may be had from, Messrs Baird & Tatlock, Glasgow.

"The foregoing method being still unsatisfactory, I was led to design the apparatus I now bring before the Society, with the view of rendering the determination of those flash-points more rapid and accurate. It is an adaptation of the Pensky-Marten slide and stirrer to the Abel's cup. Those who now employ the method recommended by the Oil Association chemists can easily have a stirring arrangement, such as I now show you, fitted to the cup. In working with the Pensky-Marten apparatus, I found the results under varied conditions to be excellent, and was struck with the idea that it could be simplified, and made more suitable for everyday work in the technical laboratory.

"The stirrers, consisting of two sets of vanes, one in the oil and the other in the vapour space, are attached to a vertical shaft passing through the cover and terminating at the top in a small bevelled wheel, the bevel of which is milled. A horizontal shaft (carried on two supports) terminates at one end with a bevelled wheel gearing with the one on the stirrer shaft, and at the other end with a disc carrying a small handle with which to rotate the shaft. This shaft also carries a collar with two pins projecting about ¼ inch at diametrically opposite points. By sliding the shaft slightly to the right, the bevel wheels are put out of gear, and the pins projecting from the collar are drawn into position for actuating the testing arrangement. This consists of a sliding cover on the top of the lid proper, so arranged that when the former is moved it depresses the pip light to the proper point, and brings the orifices opposite those on the fixed lid. There are three orifices, one, at which the test light is applied, the other two, situated one at either side of it, being for the admission of air. It will thus be readily seen that the change from stirring to testing and *vice versa* can be instantly performed.

"The results got by this apparatus are identical with those got by the Pensky-Marten. They are not affected by the source of heat, a naked flame giving the same results as the sand-bath. It is immaterial what the rate of heating is, provided it is not too rapid to prevent a correct reading of the thermometer. I prefer to reduce the rate of heating when nearing the point at which the oil is expected to flash. The stirrers do not require to be worked continuously but merely at intervals. I find it advisable to stir more frequently when the rate of heating is fast."

4. **Setting-Point.**—The determination of the setting-point or melting-point of crude oil is necessary not only as an indication of the amount of solid paraffin it contains but as a guide to the condition it is likely to assume during transport, if exposed to low temperatures. Some crude oils are so rich in solid paraffin that they solidify at temperatures between 70° F. and 80° F. Pumping is therefore impossible if the oil is allowed to fall below that point. The most suitable test for this purpose is that known as the flow test, carried out in the following manner :—

About 1½ inches of the oil to be tested are poured into a ⅝ths test-tube. A thermometer is inserted into the oil, and the whole immersed in a feezing mixture until the oil is quite solid. The tube is then removed and held in a horizontal position, without stirring. As soon as the frozen oil melts from the bulb of the thermometer the temperature is noted as flow-point.

For determining the setting-point of fractions obtained from a distillation of the crude oil or of finished intermediate or heavy oils, the test is that set forth by Sir Boverton Redwood :—[1]

" Into a test-tube having a diameter of about 1¼ inches, the oil to be tested is added to the depth of about 2 inches ; the tube is then immersed in a freezing mixture, the oil being stirred with a thermometer till it is cooled down considerably below the temperature at which the solid paraffin first appears. The tube is then removed from the freezing mixture, the oil constantly stirred with the thermometer, and the point carefully watched at which the last trace of paraffin disappears. This operation is repeated with the same sample of oil until two experiments give concordant results, the temperature so found being the setting-point."

The setting-point of the heavier fractions from the crude oil, or the melting-point of crude paraffin scale or finished wax, may be approximated by simply noting the temperature at which the melted oil solidifies on the bulb of a thermometer. For the exact determination of melting-point of wax the following method[2] is generally employed in the British oil trade :—" This is determined by what is known as the 'English' test—*i.e.* a test-tube about 1 inch in diameter is filled to the depth of about 2 inches with the

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[1] Redwood's *Treatise on Petroleum*, 3rd Ed., vol. ii. p. 312.

[2] *Loc. cit.*, p. 308.

melted paraffin, a small thermometer is inserted, and the whole steadily stirred, while the test-tube and its contents are allowed to cool slowly. The temperature at which the thermometer remains stationary for a short time is the melting- (setting-) point."

The reason for the temperature remaining stationary at the point of solidification is the latent heat of fusion being given out by the wax as it changes from the liquid to the solid state.

5. ESTIMATION OF BENZINE (PETROL IN VARIOUS GRADES), KEROSENE, AND HEAVY OIL FRACTIONS.

This is effected by means of fractional distillation, and the standard method is that of Professor Engler,[1] by which the crude oil is divided into three fractions.

(1) That distilling up to 150° C., representing the amount of light spirit (petrol and benzine) contained in it.

(2) That distilling between 150° C. and 285° or 300° C., representing its yield of illuminating oils.

(3) Residue boiling above 285° or 300° C., representing intermediate and heavy oils, solid paraffins, and residue.

In the above reference Professor Engler's system of distillation is described as follows :—" For the fractional distillation of petroleum Engler employs a globular flask (fig. 17) 6½ cm. in diameter, with a cylindrical neck 1·6 cm. in internal diameter and 15 cm. in length, from the side of which a vapour-tube 10 cm. in length extends at an angle of 75° downwards to the condenser. The junction of the vapour-tube with the neck of the flask should be 9 cm. above the surface of the oil when the flask contains its charge of 100 c.c. of oil. The observance of the prescribed dimensions is considered essential to the attainment of uniformity of results. A thermometer is fixed in the cork inserted in the mouth of the flask so that the top of the bulb is on a level with the vapour-tube. 100 c.c. of the oil is introduced into the flask by means of a pipette, and heat is applied by

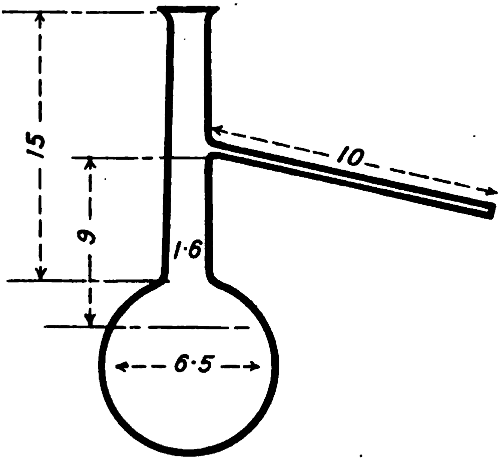

Fig. 17.—Engler distillation flask.

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[1] Redwood's *Treatise on Petroleum*, 3rd Ed., vol. ii. p. 204.

means of a Bunsen burner. At first wire gauze is interposed between the burner and the flask, but afterwards the naked flame is employed, the heat being so regulated that from 2 to 2½ c.c. of distillate passes over per minute. In this way fractions differing from each other in boiling-point by 50°, 25°, or 20° C. can be obtained. As soon as the requisite temperature (150° C. for the first fraction) is attained, the lamp is withdrawn until the temperature has fallen at least 20°, when the oil is reheated to the boiling-point and again cooled, this process being repeated until no more distillate is obtained. The oil is then heated up to the boiling-point, and the cooling and reheating process repeated, and so on with the other fractions. These precautions must be observed in order to produce trustworthy results, which (it is claimed) can be obtained with not more than a variation of 1 per cent. by this method, even in the hands of different experimenters.

"In a personal communication addressed to the author with reference to this process, Dr Engler says :—'We estimate the proportion of burning oil from the amount of the fractions between 150° and 300° C. in the case of oils consisting of hydrocarbons of the methane series (C_nH_{2n+2}), such as those from Pennsylvania, Galicia, Elsass, etc., or from the proportion of the fractions between 150° and 285° where the hydrocarbons belong to the naphthene series, as is the case with the Baku, Java, and (partially) the Burmese oils. As in practice some of the fractions below and exceeding the foregoing limits are included as burning oil, the proportion obtained on a manufacturing scale is greater than that yielded by a test-sample, but these limits give the nearest indication of the proportion of burning oil. A universal standard cannot, however, be established, because in some oils, and notably those from Baku, the fraction between 285° and 300° is so heavy as to prejudicially affect its capillary properties with regard to the lamp-wick. On the other hand, the same fraction in the American oils may be used for burning without seriously affecting the burning quality. We find inferior American oils contain as much as 15 per cent. by volume of fractions boiling below 150°, and 25 per cent. (volume) boiling over 300°, so these should be considered the extreme limits.

"'If it is only a question of determining the amount of spirit, burning oil, etc., identical apparatus and method of working are, in my opinion, of greater importance than the complete separation of the single fractions. My apparatus, now generally used in Germany, was designed with this object, and I discarded a dephlegmator as giving less regular results than a simple instrument, in the hands of different experimenters. By carefully following the directions, the margin of error is only about ¼ to ½ per cent. At present we fractionate in stages of 25° ; for instance, the benzine fraction boiling at, say, 86°, would be included in the fraction boiling at 100°, and the subsequent fractions are taken at every 25° (100° to 125°, 125° to 150°, 150° to 175°, ... 275° to 300°. By adding together the fractions up to 150°, the quantity of light spirit is ascertained ; the burning oil comprising those between 150° and 300°, and so on. The former practice of taking fractions at every 20° is discontinued. However, in estimating the actual component parts of an oil, smaller fractions are necessary. In the burning oil, the greater the proportion of the first fraction above 150° the better, the converse being true in the case of the fractions above 250°.' "

6. ESTIMATION OF SULPHUR AND CALORIFIC VALUE.

For the determination of the latter a calorimeter is required. For the former, the bomb of that instrument may be used for all oils, but for the lower boiling fractions, from intermediate oil to petrols, the lamp method has been found accurate and convenient. The directions given hereafter (page 52) for using the Mahler bomb are taken from *The Laboratory Book of Mineral Oil Testing*,[1] the diagram and reference letters being modified to suit the design of instrument illustrated.

The modification of the lamp method for the estimation of sulphur is described by Drs A. E. Dunstan and F. Thole as follows :—

The Estimation of Sulphur in Motor Spirit and Kerosene.—The methods available for the estimation of sulphur in oils are two in number. Methods have been suggested based on oxidation of the sulphur compounds by nitric acid, but they are now generally recognised as unreliable.

(1) The bomb calorimeter method, in which a definite amount of oil is burned almost instantaneously in a strong steel vessel filled with compressed oxygen, the sulphuric acid produced being weighed in the form of barium sulphate.

(2) The lamp method, which, under a variety of forms, consists in burning a known amount of oil in a small lamp and aspirating the products of combustion through an alkaline-absorbing medium in order to retain the oxides of sulphur produced. These are ultimately converted into sulphuric acid and estimated volumetrically or gravimetrically.

Each method has decided advantages and disadvantages.

The bomb method is applicable to all oils, and is on the whole very accurate, though as the amount of oil usually burned rarely exceeds one gramme, the amount of barium sulphate obtained from

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[1] *The Laboratory Book of Mineral Oil Testing*, by Jas. A. Hicks (C. Griffin & Co., Ltd.).

an oil of very low sulphur content is sufficiently small to be influenced to a comparatively high degree by experimental errors. The apparatus is, however, expensive initially; it requires a supply of compressed oxygen, and manipulative difficulties are considerable.

The lamp method is limited to distillates such as motor spirit, benzol, kerosene, and gas oil, being useless when applied to asphaltic oils such as most crude petroleums and shale oils. The apparatus is, however, simple in construction and in manipulation, while the accuracy of the method is at least equal to that afforded by the bomb method. Moreover, with oils of very low sulphur content a considerable quantity of oil may be burned and the influence of experimental errors correspondingly reduced.

It will be obvious that the products of combustion in the lamp method contain oxides of sulphur in a very high degree of dilution, and the accuracy of the method will therefore depend primarily on the efficiency of the absorbing apparatus employed.

In most forms of the lamp apparatus this has consisted of a column of moistened glass marbles (Gas Referees' apparatus) or glass beads, or of a series of ordinary gas-washing bottles. The efficiency of the first type is very doubtful, while the other systems are open to several objections. We have therefore devised a form of absorber, which is simple in construction and highly efficient in action (fig. 18).

The gases are broken up by impinging on a series of short lengths of glass tubing, and subjected to a very thorough scrubbing action. At the same time the apparatus opposes much less resistance and can therefore deal with a more rapid current of gas than a bead-filled washer of equal size.

Method of Procedure.—A suitable quantity of the oil (usually 10 c.c., but less if a considerable proportion of sulphur is present) is measured or weighed into the lamp, which consists of a 1-oz. bottle fitted with a cork carrying a wick-holder of silica tube 1¼ inches by ¼ inch; the wick is ordinary loose cotton. If the oil is measured, its specific gravity should be known in order to express the result as percentage by weight. The oil is dissolved in two to three times its volume of absolute alcohol; if alcohol does not dissolve the oil readily, a little amyl acetate may be added.

In the flask, F, which stands in a beaker of water and serves principally as a cooler for the hot gases, is placed about 3 c.c. of sodium hypobromite. This solution is made by dissolving 10 gms. of sodium carbonate and 4 c.c. of bromine (washed with water) in 200 c.c. of cold distilled water.

In the washers, W, is placed a little water (about 1 inch depth), and ½ gm. of solid sodium peroxide.

The products of combustion are drawn through the apparatus by a water pump of fairly large capacity or, more conveniently, by a Lennox blower, the speed of which should be controlled by a lamp or wire rheostat.

The flame of the lamp should be smokeless; if smoke is produced, the air-current should be increased or the size of the flame reduced by lowering the wick. When all the oil has been burned the lamp is rinsed by burning three successive 2 c.c. amounts of alcohol. The burning usually takes 2½ to 3 hours.

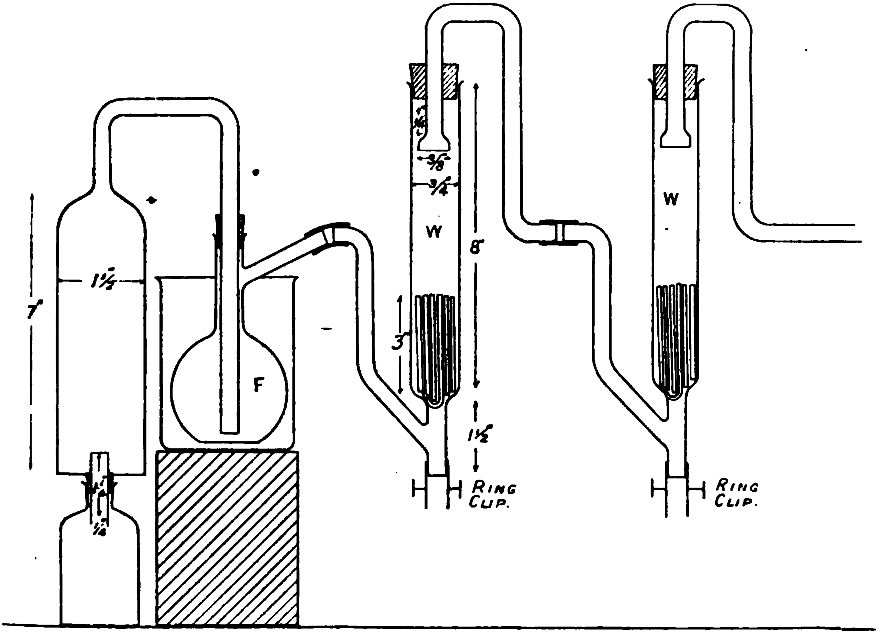

FIG. 18.—Modified lamp-test apparatus for sulphur.

The contents of the absorption vessels are then transferred to a beaker, acidified with hydrochloric acid which has been freed from sulphates by adding a little barium nitrate; barium nitrate is added, and the liquid boiled and then allowed to stand for not less than 15 hours. The barium sulphate is then filtered off, washed, and ignited. The ignited product is treated with a drop of dilute sulphuric acid, reignited, and finally weighed. It has been found that even the purest obtainable reagents contain traces of sulphates, and it is therefore necessary to carry out a blank determination whenever a fresh stock of reagent is used. If a reasonable stock of reagents is made up, it should last for several months. The variation in the sulphur content of the laboratory air is, as a rule, negligible, but if any considerable variation is suspected, a parallel blank experiment should be made.

TABLE V.—COMPARATIVE TESTS OF THE BOMB AND LAMP METHODS.

Material.	Sulphur (Bomb).	Sulphur (Lamp).	Average (Lamp).
Refined petrol	·03	·03 ·03 ·03	·03
Kerosene, Sample A . . .	·16	·17 ·16 ·18 ·16 ·18	·17
Kerosene, Sample B	·22	·20 ·20 ·21 ·21 ·20 ·21 ·20	·204
Mexican gas oil	1·66	1·64 1·65 1·65 1·66	1·65
Shale gas oil	1·29	1·30	. .
Alcoholic solution of ethyl disulphide containing ·261 per cent. sulphur.	. .	·265 ·261 ·267	. .

"**Estimation of Sulphur.**—After numerous experiments carried out under the instructions of Sir Boverton Redwood, with very varied methods, it is clearly evident that while one method may be suited to one class of oil and another to another class, the bomb calorimeter is the most trustworthy apparatus for employment with all oils. Full details as to the working of this instrument are given below.

"After the ignition of the sample as there prescribed, the bomb is connected by a piece of rubber tubing to the bottom of a cylinder of glass beads moistened with weak alkali solution (free from sulphur), and the gaseous products of combustion are washed free from sulphates before escaping into the air. The bomb is opened, the washings poured into it, and then through a quick filter into a beaker, together with three or four rinsings with distilled water. The usual precipitation with barium is then carried out.

"**Calorific Value.**—The question of the thermal efficiency of oils is occupying the attention of the petroleum specialist to an increasing extent in view of the steadily extending use of liquid fuel for industrial purposes. The apparatus yielding the most satisfactory results with a minimum of trouble is undoubtedly some form of "bomb calorimeter," such as that employed by M. Berthelot. The high cost of this particular instrument, owing to the amount of platinum used in its construction, often places it beyond the reach of the chemist, but a very good substitute has been designed by Mahler, and may be safely recommended.

The following free translation from the French of the pamphlet supplied by the makers gives full guidance as to its manipulation :—

"The calorimeter (fig. 19) is essentially composed of a bomb, B; a calorimeter, D; an insulating jacket, A; and an agitator, S. The bomb has a capacity of about 650 c.c., and the walls are 8 mm. thick. This capacity assures complete combustion of the sample by providing a decided excess of oxygen.

"The bomb, made of specially forged steel, is nickel-plated outside, and has an inner coating of enamel[1] to resist the corrosive action of the nitric acid, which always forms during the combustion. The bomb is closed by a screw lid fitting tightly on a lead washer. The cover carries a screw valve, which permits the introduction of oxygen.[2] Through the cover, or lid, a platinum electrode, E (well insulated), is carried into the interior of the bomb.

"Another rod of platinum is also fixed, supporting the capsule plate, E_1, where the sample to be tested is placed.

"The sample is ignited by contact with the iron wire spiral, F (of known weight), which is connected at the desired moment to an electric current of about 12 volts and 2 amperes. A simple mechanical combination works the helicoidal agitator, and allows the operator to give a regular stirring movement without trouble. On the right side of the stand carrying the manometer is placed a screw valve, which can easily be adjusted to allow the slow introduction of the oxygen into the bomb. This valve avoids the necessity of using the valve on the oxygen cylinder, and is much more easily adjustable to give a slow supply of oxygen. M. Mahler uses the cylinders of compressed oxygen as figured, and, as a suitable pressure for the combustion of 1 gramme of oil is 25 atmospheres, a cylinder containing 1000 litres of gas at 120 atmospheres would be sufficient for about sixty experiments.

"Weigh 1 gramme of the substance to be tested into the capsule, E_1; adjust the iron wire spiral used for igniting the sample. After the capsule has been firmly fixed on its supporting rod, screw on the

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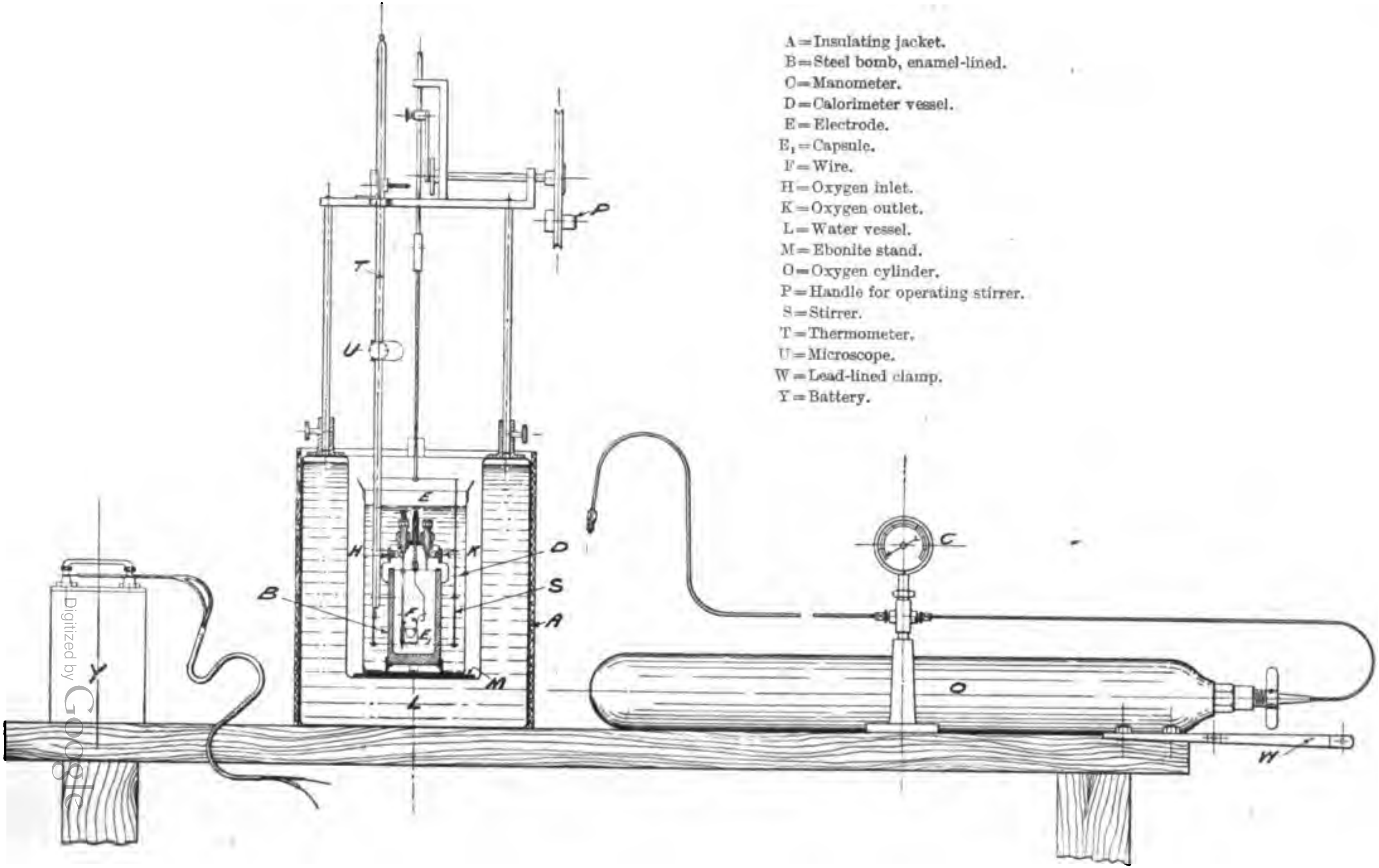
[1] The specific heat of steel is 0·1150; this specific heat has been determined by M. Matignon, at the College of France (laboratory of M. Berthelot). The specific heat of enamel is 0·2045; also found by M. Matignon.

[2] The small nut on the top of the milled head is provided to keep the screw valve in position by means of a spanner when the tube is screwed on.



A = Insulating jacket.
B = Steel bomb, enamel-lined.
C = Manometer.
D = Calorimeter vessel.
E = Electrode.
E_1 = Capsule.
F = Wire.
H = Oxygen inlet.
K = Oxygen outlet.
L = Water vessel.
M = Ebonite stand.
O = Oxygen cylinder.
P = Handle for operating stirrer.
S = Stirrer.
T = Thermometer.
U = Microscope.
W = Lead-lined clamp.
Y = Battery.

FIG. 19.—Sketch of Mahler bomb calorimeter.

lid of the bomb very firmly by gripping the latter in the vice provided, and using the large spanner. Connect the screw valve of the bomb to the tube leading from the pressure gauge, taking care to have the screw valve on the manometer stand closed. First open the tap of the oxygen cylinder, O, and then very gently open the screw tap on the manometer stand and allow the gas to flow until it registers a pressure of 25 atmospheres on the manometer. Now close the manometer screw tap, the screw tap of the oxygen cylinder, also the screw tap on the cover of the bomb, and disconnect the tube from the bomb. It is recommended that the substance to be tested should not be in too fine a powder, as the current of gas entering may blow it out of the capsule. To avoid this, care should be taken to allow the oxygen to enter the bomb slowly.

"Place the bomb in the calorimeter, D, then put the thermometer, T, and the agitator, S, in position, and pour in the water, which has been previously measured. Leave the apparatus so assembled for a few minutes, stirring at intervals so as to ensure a somewhat equable temperature before commencing observations. Then, when all is ready, the stirring is performed continuously, and a careful note taken of the temperature every minute for about five minutes, so as to fix the law which the thermometer follows before the substance is ignited. Now ignite the substance by connecting one wire of the battery to the platinum electrode, E, and the other wire to some part of the screw tap. Take note of the temperature half a minute after the ignition, also at the end of this minute, and continue to take the thermometer readings every minute until they commence to show a regular fall in temperature. This is the maximum temperature to be noted. Continue the readings of the thermometer for another five minutes, so as to fix the law the thermometer follows after the maximum temperature has been reached."

"The following are the rules for correction of calculation:—(1) The law of the decrease of temperature observed after the maximum represents the loss of heat of the calorimeter before the maximum for any one minute, on the condition that the average temperature of this minute does not differ by more than one degree from the maximum temperature. (2) If the temperature of the examined period differs by more than one degree, but by less than two degrees, from that of the maximum, the figures which represent the law of decrease at the moment of the maximum diminished by 0·005 give the correction required. The two preceding remarks suffice in all cases. Moreover, without affecting the precision of the experiment, it may be taken that the law of variation followed during the first half of the minute during which the ignition took place is that which existed at the beginning of that minute. During the whole time of the observation the operator ought to take care to see that the agitator acts regularly.

"When the observation is finished, first open the screw tap, then the bomb itself. Now wash the interior of the bomb with a little water, so as to collect the acid formed during the explosion. The nitric acid is estimated by titration with alkali, and now we are in possession of all the elements for the calculation, for since the calorific power, Q, is the whole—

$$Q = \Delta(P + P') - (0{\cdot}23p + 1{\cdot}6p').$$

Δ = the difference of the corrected temperature.
P = the weight of water in the calorimeter.
P′ = the equivalent of water in the bomb and its accessories.
p = the weight of the nitric acid (HNO_3) found.
p' = the weight of the small iron spiral.
0·23 = the heat caused by the formation of 1 gramme diluted nitric acid.
1·6 = the heat caused by the combustion of 1 gramme of iron.

"If it is a question of testing oil, no notice need be taken of the small quantity of sulphuric acid which results from the oxidation of the sulphur of the sample, and which will be estimated in the titration as nitric acid. The error is in reality negligible in a commercial estimation. But one will notice that the bomb gives a means of estimating the sulphur, which is entirely oxidised and transformed into sulphuric acid.[1] In this case it is unnecessary to take note of the thermometer reading. In the case of a test of a substance with little hydrogen in it—coke, for example,—so little water is formed that the quantity is insufficient to dissolve the acids. It is then necessary to put at the bottom of the bomb a few c.c. of water, which one must take into account in the calculation.

"Proceed just the same for a liquid as for a solid. But if the liquid emits appreciable vapours at ordinary temperatures, it is better to weigh the amount taken in a thin capsule with slender points, through which passes the fuse of iron wire. When the capsule is introduced into the bomb, care must be taken to break these points, so as to allow the oxygen to make contact with the liquid.

"The method of finding the calorific power of gases is as follows:—Carefully measure the empty space in the bomb; fill it first of all with gas, now empty and definitely introduce the gas under atmospheric pressure at the temperature of the laboratory, then add the oxygen, and proceed the same as for solids or liquids.

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[1] The heat given out by the formation of the diluted sulphuric acid, H_2SO_4, can be calculated on the basis of 0 cal. 73 per gramme of H_2SO_4.

"The determination of the calorific power of gases offers a peculiar difficulty. Care must be taken not to mix the gas with such a quantity of oxygen that the mixture ceases to be explosive. For ordinary lighting gas 5 atmospheres of oxygen are sufficient. For the gas of Siemens' generator not more than half an atmosphere, measured by the mercury manometer, must be taken.

"**Determination of the Equivalent in Water of the System.**—To find the term of correction representing the exact equivalent P′ of the system in water, the most simple way is to make a double experiment, as follows:—Burn in the bomb a known weight, 1 gramme, of naphthalene for example, with 2300 grammes of water in the calorimeter. Next burn 1 gramme of naphthalene with only 2100 grammes of water in the calorimeter. We have, then, two equations which between them eliminate the calorific value of the naphthalene, and the water equivalent can be deduced from the difference in the two sets of figures. Care must be taken to weigh the naphthalene after it is half melted; otherwise, if it were not fused, some of it would be scattered by the current of oxygen and would not be burnt.

"**Example of the Determination of Calorific Value.**—The combustible was a sample of colza oil. Its analysis gave—carbon, 77·182 per cent.; hydrogen, 11·711 per cent.; oxygen and nitrogen, 11·107 per cent. The weight taken was 1 gramme. The calorimeter contained 2200 grammes of water. The equivalent in water of the bomb and its accessories was 481 grammes.[1]

"The apparatus having been assembled as described, a few minutes were allowed to elapse, so as to establish a uniform temperature, the stop-watch was started, and the thermometer readings recorded as below:—

PRELIMINARY PERIOD.

0 minute	10·23°
1 ,,	10·23°
2 minutes	10·24°
3 ,,	10·24°
4 ,,	10·25°
5 ,,	10·25°

$$\Delta_o = \frac{10{\cdot}25° - 10{\cdot}23°}{5} = 0{\cdot}004$$

ignited by means of electrodes.

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[1] The equivalent in water, 481 grammes, was found by a special method giving directly the exact heat value of the system.

PERIOD OF COMBUSTION.

5½ minutes	10·80°
6 ,,	12·90°
7 ,,	13·79°
8 ,,	13·84° (maximum)

LAST PERIOD.

9 minutes	13·82°
10 ,,	13·81°
11 ,,	13·80°
12 ,,	13·79°
13 ,,	13·78°

$$\triangle T = \frac{13{\cdot}84^\circ - 13{\cdot}78^\circ}{5} = 0{\cdot}012.$$

"The thermometrical observations were stopped. The variation of the temperature was—

$$13{\cdot}84^\circ - 10{\cdot}25^\circ = 3{\cdot}59^\circ.$$

"Take note of the following corrections. The system had lost, during the minutes (7, 8) (6, 7), a quantity of heat =

$$\frac{13{\cdot}84^\circ - 13{\cdot}78^\circ}{5} \times 2 = 0{\cdot}012 \times 2 = 0{\cdot}024.[1]$$

During the half-minute (5½, 6) a quantity of heat

$$= (0{\cdot}012 - 0{\cdot}005)\ \tfrac{1}{2} = 0{\cdot}0035,$$

and during the half-minute (5, 5½) it gained

$$\frac{10{\cdot}25^\circ - 10{\cdot}23^\circ}{5} \times \tfrac{1}{2} = 0{\cdot}004 \times \tfrac{1}{2} = 0{\cdot}0020.[2]$$

It follows that the net loss during the minute (5, 6) was $0{\cdot}0035 - 0{\cdot}002 = 0{\cdot}0015$.

"Altogether the system lost during the experiment a quantity of heat $= 0{\cdot}024 + 0{\cdot}0015 = 0{\cdot}0255$, which must be added to the 3·59° already found.

"The variation of the corrected temperature is then 3·615°, neglecting the fourth place. The quantity of heat observed is therefore $(2{\cdot}200 + 481) \times 3{\cdot}615 = 9$ cal. 691815. Take 9 cal. 6918.

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[1] Law of cooling after the maximum.

[2] Law of variation of temperature at the moment of the minimum.

"To get the required result we must subtract from this :

(1) The heat caused by the formation of 0·13 gramme of nitric acid, HNO_3, titrated volumetrically, 0·13 × 0·23 =0 cal. 0299

(2) The heat of the combustion of 0·025 gramme of iron thread, 0·025 × 1·6 =0 cal. 0400

To be subtracted 0 cal. 0699

The final result is then

9 cal. 6918 – 0 cal. 0699 = 9 cal. 6219,

or for 1 kilogramme of oil, 9·621 cal. 9.

"Note that the dimensions of the apparatus are such that one can, without difficulty, arrange once for all so that in all experiments the insignificant corrections cancel one another. If x is the correction due to loss of heat during the operation, it will not be necessary to make the correction if

$$0{\cdot}23p + 1{\cdot}6p' = x\,(P + P').$$

p' is at the disposition of the worker within certain limits ; P equally so. Evidently, then, it is possible to make the equation sufficiently true. Thus the calorific value of colza oil is 9621·9 cal. Simply multiplying 3·59 by 2681 gives 9624, which approximates to within 2 in 9000."

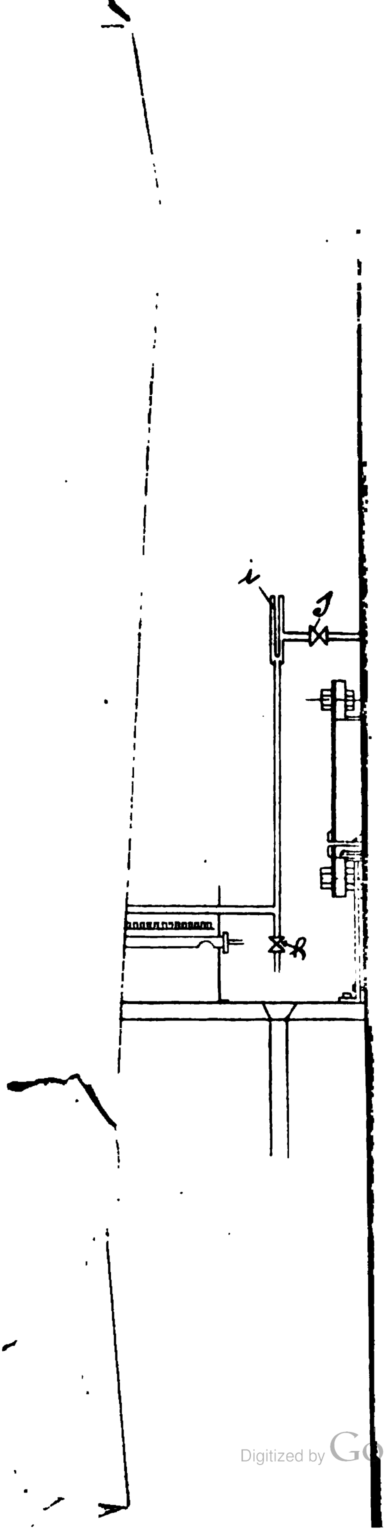
7. **Fractional Distillation of the Crude Oil in 5 per cent. Fractions.**—In order to formulate a complete refining process it is necessary to carry out that process in miniature, experimenting with representative samples of the oil to be treated on the large scale. The results obtained will enable the refiner to design and arrange the whole plant to the best advantage. A still having a capacity of 5 gallons is desirable, for that quantity can be completely distilled and the fractions carefully measured within usual laboratory working hours. To ensure sufficient of the various fractions for further examination, several distillations should be made and the fractions bulked together. In this way ample oil will be obtained to enable all tests of the fractions from the crude oil to be performed, second stage operations worked in miniature, and samples of intended products prepared. Thus a valuable insight into the working of the process will be found, and much trouble avoided when operations on the large scale are commenced.

The experimental still may be either of the "pot" or "cylindrical" type, heated preferably by gas burners. For crude oil distillation, *i.e.* the first stage in the operation, the author prefers a plain still-head attached to the still. A condenser of adequate cooling surface is connected, and arrangements made for the control of the condensing water to prevent solidification of paraffin, should any exist, in the heavier fractions distilling over. This is done by checking the flow of water, stopping it, or even running it off, to allow the temperature of the condenser to heat up to the point necessary for the distillates always to remain liquid. The determination of the setting-point of the crude oil itself and that of the residue from the Engler distillation will indicate what is to be expected when the heavy fractions distil over.

The condenser should be connected to a vacuum pump to allow of the distillation being conducted, entirely or partially, under diminished pressure, a very necessary precaution if the oil under examination is prone to "cracking," which, if allowed to take place, will lower the flashing-point and be detrimental to viscosity, colour, and yield of paraffin of the heavy distillates.

The still is connected to a steam generator and the steam therefrom passed through a superheating furnace. After the benzine has distilled off, the rate of distillation is maintained by introducing steam into the still as well as by increasing the fire heat. The temperature of the steam at this stage is only sufficiently high to ensure it being quite "dry," but it may be advantageously increased in temperature as the distillation progresses. The volume of steam, measured as condensed water passing over with the distillate, will vary from a small quantity at the commencement, increasing towards the end. The average may be taken as 25 per cent. of the condensed distillate.

Fig. 20 illustrates an experimental still of the type described, which has been found to give excellent results. The still-head is interchangeable with a dephlegmator for the refractionation of the crude benzine, *i.e.* all the fractions of the crude oil containing appreciable quantities boiling under 150° C.

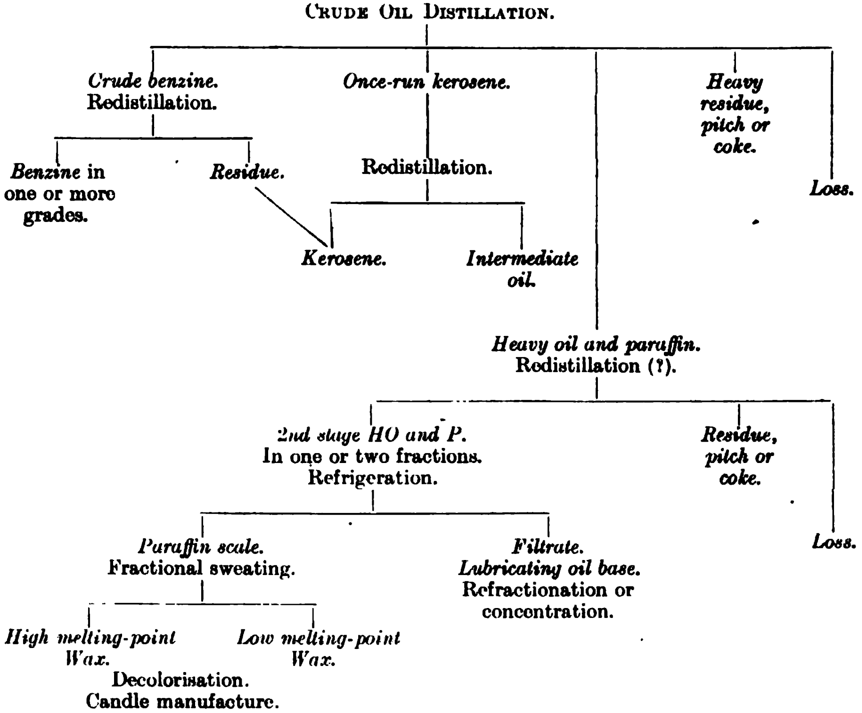

i
j
k

Explanatory Key to Fig. 20.

A	**Boiler,** capacity 10 gallons.
a	Gauge glass with drain cock to gutter.
b	Steam gauge, to 40 lbs. per sq. in.
c	Safety valve to blow off at 15 lbs. per sq. in.
d	Filling cock from water main.
e	Blow-off cock delivering into drain connecting with gutter.
f	Three - ring Fletcher burner.
B	**Steam superheater.**
g	Fletcher burner.
h	Drain cock for surplus steam and condensed water; delivering into drain connecting with gutter.
i	Thermo-tube, for registering temperatures of steam.
C	**Still,** capacity 5 gallons.
j	Cone-valve for regulating steam supply to still.
k	Steam distributor, perforated with $32 \times \frac{1}{16}$ inch holes, staggered at 45° from vertical.
l	Thermo-tube for registering temperature of oil in still.
m	Drain cock.
D	**Dephlegmator,** to replace still-head when desired.
q, *q*	Unions connecting still-head and dephlegmator to condenser.
E	**Condenser.**
r	Water inlet to condenser.
s	Water outlet from condenser.
t	Condenser support.
u	Union connecting condenser to worm-end.
F	**Worm-end.**
v, *v*	Worm-end cocks with taper connectors; receivers attached by rubber corks.
G	**Still-head.**
n	Thermo-tube for registering temperature of oil vapours.
o	Filling cock.
p	Three Fletcher's burners in parallel.
H	**Vacuum water pump.**
w	Connection to vacuum pump.
x	Vacuum trap and manometer.
y	Mercury back pressure safety trap.
z	Water-supply cock to pump.
z′	Compo-tubing allowing fall of 20 feet to water coming from pump.

The following diagram is illustrative of the full process, which, of course, may be modified to suit the oil to be dealt with. For instance, the kerosene fraction, at the once-run stage, may be suitable for market conditions without redistillation, or the heavy oil may contain no solid paraffin, in which case refrigeration will be omitted. There may be a good market for fuel oil, and, if so, it may be best, from a financial point of view, simply to remove the benzine and kerosene fractions, or even the benzine only, leaving the residues therefrom available for disposal as fuel oil. Therefore it will, as already stated, be necessary to adopt the full refining process illustrated, to the analysis of the crude material and to market conditions existing for products. For larger concerns, of course, with facilities for transport, the whole world is practically open as a market for products; but the lesser, with limited resources, may be confined to a small local field for the disposal of produce. All these points must be considered by the refiner in formulating the process of manufacture.

DIAGRAM 1.—FULL REFINING SCHEME.



The 5 per cent. fractions are tested individually and the results tabulated. Specific gravity, flash-point, setting-point, and fractional composition are determined and recorded. It is a good plan to begin the flash-points on the last fraction over with the Gray tester, and work back until the result comes within the range of the Abel instrument, carrying on therewith until reaching the fraction whose flash-point is too low for determination at all. In the same manner the setting-points should be examined, testing those that may be solid by the bulb test, and changing to the test-tube method when the amount of solid paraffin diminishes, and ceasing when a fraction is discovered with a setting-point about 15°–20° F.

For the examination of the fractional composition of the 5 per cent. fractions the author uses Sir Boverton Redwood's method for the examination of petrols. This is applied until the intermediate fraction is reached and the determination of initial and final boiling-point omitted.

The method above referred to is as follows :—

" 100 c.c. of the sample are measured into a Wurtz distillation flask, 2¾ inches in diameter, with a neck ¾ inch internal diameter, the distance from the shoulder, or top, of the globular portion to the lateral exit tube for vapour being 2½ inches. The angle between the exit tube and neck should be 75 degrees.

" A perfectly sound cork, bored quite centrally, is fitted into the neck of the flask, and a cylindrical-bulbed thermometer is passed through the hole in the cork so that the bulb is half immersed in the liquid contained in the flask.

" The flask and contents are placed on wire gauze, and the exit tube of the flask connected to a Liebig's glass condenser, the inner tube of which is 24 inches in length. (Before the experiment is carried out the interior of the condenser tube is wetted with a little of the sample to be treated, and then allowed to drain.)

"A small flame is first placed under the wire gauze and the temperature of the liquid gradually raised.

" When vapour is first evolved it will be seen to condense in the sides of the flask just above the surface of the liquid, and as the temperature is increased the vapour will be seen rising into the neck of the flask. While this is taking place, the thermometer is raised to follow the vapour until the centre of the bulb of the thermometer is 1 inch below the exit tube, care being taken to keep the bulb completely surrounded by vapour. When the first drop of condensed vapour falls from the end of the exit tube of the flask into the condenser, the temperature of the vapour, as shown by the thermometer, is noted, and this temperature is recorded as the initial boiling-point.

" After the initial boiling-point has been observed the size of the flame is increased, so that the rate of distillation is 2 to 2½ c.c. per minute, and the percentages of distillates noted at various temperatures as required, the thermometer still being in the position mentioned above (*i.e.* 1 inch below exit tube).

"The final boiling-point is the temperature observed when the bottom of the flask becomes dry. In some cases, at this point, a drop of condensed vapour falling from the bulb of the thermometer on to the dry surface is immediately converted into dense vapour resembling smoke."

If the results obtained by carrying out the foregoing seven points for determination are tabulated and considered in connection with market requirements, subsequent stages of treatment may be decided upon as found necessary.

It might be decided to separate the benzine fractions only, disposing of the residue as fuel oil, and in that case, it will only be necessary to consider the composition of the distillates to be removed and the physical properties of the residue. If kerosene is to be produced in the maximum quantity yielded by the crude material, the treatment required to render the crude oil distillates suitable for the intended market, and the character of the remaining residue, must be considered. In the event of full refining operations being advisable, to produce the maximum yields of benzine, kerosene, intermediate and heavy oils, paraffin, if any, and pitch, coke, or heavy residue, it will be necessary to continue the examination further, as follows :—

Benzine Fractions.—All the 5 per cent. fractions containing more than a few per cents. boiling under 150° F. should be bulked together, and 2 litres distilled over a good standard fractionating column, or dephlegmator, into small fractions. Whether these measure 2 per cent. or a larger quantity will depend on the problem to be considered. Since, in this case, full refining is intended, where it is well to know the exact fractional composition of these fractions which represent the crude benzine of large operations, it is best to divide the distillate into 2 per cent. portions. The temperature at which each distils into the condenser, and the specific gravity, must be noted and tabulated. In this way the disposition of the fractions to various marketable products will be readily made, and the crude benzine split up into petrols, heavy benzine, white oil (turpentine substitute), and kerosene, the results approximating very closely to those obtainable on the large scale by modern plant.

Figs. 21, 22, 23 show three forms of fractionating columns, viz. the 12-pear, the Glinsky, and the Le Bel-Henniger. There are other forms of fractionating columns, particulars of which will be found in works of reference on the subject. Those mentioned are instruments largely used for this class of work. In actual practice the amount of crude benzine to be redistilled, or rectified, may be reduced to the minimum by introducing a dephlegmator between the crude oil still and condenser.

Kerosene Fractions (once-run kerosene) may require redistillation, either wholly or partially. That is, some, the lighter, fractions, may be passed to the second stage direct from the crude oil distillation, while the heavier only require redistillation to separate fractions boiling above the kerosene limit. The fractional examination of the 5 per cent. fractions will reveal what is necessary in this respect, and the operation may be performed in a still fitted with an ordinary still-head. Marketing opportunities will, of course, be the deciding factor for the operations to be carried out.

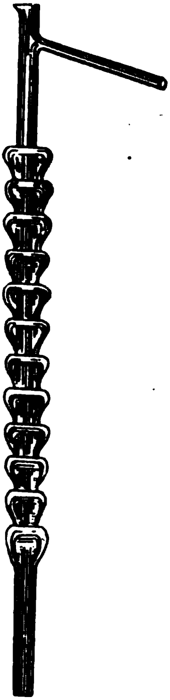


FIG. 21.—12-pear dephlegmator.

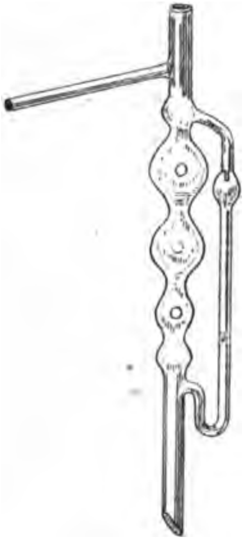


FIG. 22.—Glinsky dephlegmator.

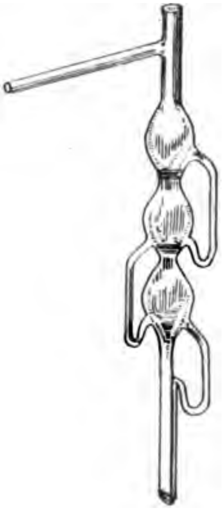


FIG. 23.—Le Bel-Henniger dephlegmator.

Intermediate Fractions existing between the kerosene limit and heavy oils may be those separated at the crude oil distillations, the heavier fractions from the kerosene redistillation, or the lighter fractions from the redistillation of the heavy oil and paraffin filtrate.

Heavy Oil Fractions.—Should these contain no solid paraffin their fractionation will be the same as that performed on the oils from which paraffin has been extracted. When they contain solid paraffin they are bulked together and the whole frozen into a solid mass. It is sometimes advantageous to include with those containing much paraffin one or more of the fractions containing no solid matter, for good crystallisation and separation of the oil from the paraffin result from such a mixture. The selected fractions bulked and frozen solid in a freezing mixture are pressed carefully and gradually in a screw press until the larger portion of the oil is expressed, the frozen mass being contained in a bag of filter cloth specially made for the purpose. The scale is then removed from the bag and repressed in another of finer texture to ensure the removal, as far as possible, of any oil remaining in it.

An ordinary letter press, or, better still, a screw tincture press, packed in ice, will be found suitable for this work. The first pressing should not be conducted in too high a temperature, a gradual rise to normal being preferable, as the objects in view are the production of (1) filtrate containing as little solid paraffin as possible when examined for set point by the test-tube method, and (2) scale containing a minimum of oil only.

A more satisfactory method of separating the scale from the heavy oil and paraffin is to employ a filter press, as illustrated by fig. 24. The use of this will enable the refiner to record useful data as regards the rate of filtration per square foot of filtering area and the character of the scale to be removed. If the oil contains little solid paraffin, the separation may be effected in one stage; but if the paraffin is abundant, it will be found more expeditious and economical to cool and filter in two stages.

The scale from both stages is mixed together in the proportion in which it exists in the oil, melted, and formed into a solid cake by allowing the melted mass to cool to the atmospheric temperature. The cake will be of a convenient size to be placed in a water-bath, and its thickness should measure above half an inch. The temperature of the water-bath will be controlled by a regulating device, and the cake will rest on a shelf of gauze fitted into a tray to receive the "sweatings." By the gradual increase of temperature the scale may thus be fractionated into various grades. In most cases coming within the author's experience the highest fraction becomes almost white by the above sweating, and requires only a final filtration through a decolorising medium to render it perfectly so. In some cases it will be necessary to "resweat" the scale to attain best colour results, or to treat the sweated paraffin with sulphuric acid before the final decolorising filtration. Several mediums are used for this last operation, charcoal, Japanese acid clay, fuller's earth, and bauxite being in regular use at the present time. Bauxite is very efficacious as a decolorising medium for wax, kerosene distillates, and for residuum lubricants.

The ore must be ground to a state of division which will give best decolorising effect, and yet not so fine that it will clog the filter. It is ignited at a dull red heat until all, or most of, the water of combination it contains is driven off. It is then placed in a tubular filter, kept at a temperature well above the melting-point of the wax to be decolorised. The "runnings" are, at first, if the wax is susceptible to the action of the bauxite, quite white, but they gradually become darker, until the efficiency of the bauxite is exhausted.

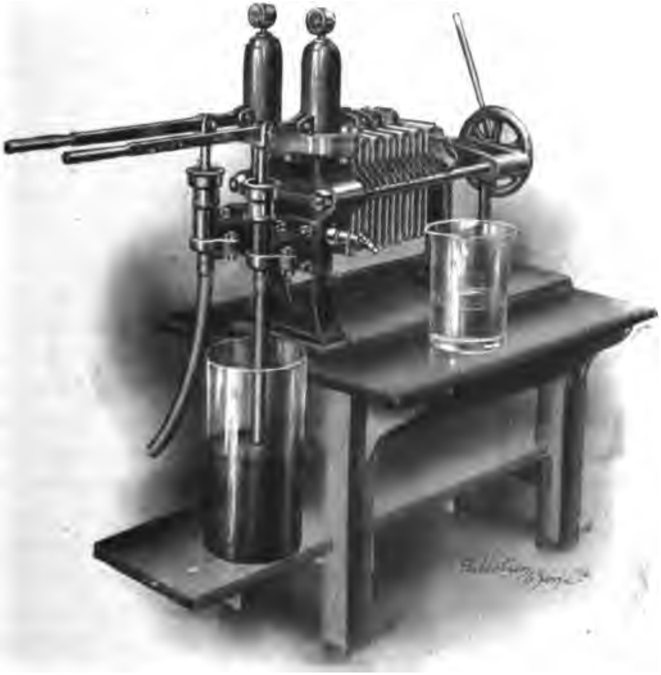


FIG. 24.—Laboratory filter press.

About 1 lb. of ignited bauxite will decolorise 1 gallon of melted wax, which must, of course, be perfectly free from water. In laboratory work the extraction and recovery of the wax adhering to the bauxite will be readily accomplished by washing with petroleum spirit or light oil. The recovery on the large scale will be explained later.

The Filtrate, after removal of the solid paraffin, must be examined to ascertain what proportion is suitable for lubricating oils. This is done in a small metal still—say 2 gallons capacity—fitted to arrangements already made for superheated steam and diminished pressure. The division of the distillates into 10 per cent. portions, and the determination of their specific gravity, flash-point, and viscosity at

Google

varying temperatures will furnish a good indication of percentage yields of oils suitable for lubrication of light and heavy machinery, and for use in electrical transformers, switches, etc. The filtrate may require treatment with sulphuric acid before redistillation to ensure good coloured distillates.

Another way of obtaining the same results is to divide the heavy oil and paraffin distilling from the crude oil into several fractions and to extract the paraffin from each separately, the tests of the filtrates being recorded.

Portion of the lubricants may be produced as residues, in which case a given quantity is distilled off the filtrate. Usually the greater the quantity taken off the higher the tests of residue left in the still. Considerable experience in the manipulation of the fire heat and superheated steam is necessary for obtaining best results. It is desirable to produce lubricants at the lowest possible temperature to obviate risk of decomposition in the still.

Pitch, Coke or Residue.—If the crude oil is worked for residue to be used as fuel for manufacturing purposes, the distillation is simply stopped at a determined point. For instance, the last distillates coming over might be very viscous and highly coloured and expensive to work into good products. If left in the still they might make good pitch, and a market might be available for coke. In that case, perhaps the most economical procedure is to leave them in the still as a residue, which is blown or run out into a cooler, whence it is transferred to the fuel tanks. When running the distillation to coke, the still is fired until the steam blows all the oil vapours from the still and condenser. When the clean steam blows through, the fire is withdrawn, allowing the steam to pass for a short time afterwards. In this way the coke is deposited in what may be called an aerated condition on the still bottom. Without this steaming the coke will be very hard and difficult to break. When the "coking" stage is reached the vacuum pump reducing the pressure on the still will, of course, be withdrawn.

When pitch is desired the operation is similar to that for making coke. A trial experiment will be necessary to ascertain what quality is obtainable, and considerable experience is required for the proper adjustment of steam and fire to produce the best results.

CHEMICAL TREATMENTS AT INTERMEDIATE AND FINAL STAGES.

So far nothing has been said about chemical treatments which may be necessary at intermediate or final or all stages if the resultant products are to be of finest quality obtainable from a crude oil yielding distillate of inferior quality. The chemicals in general use for petroleum distillates or residues are sulphuric acid, caustic soda, and litharge.

If the crude oil is non-sulphurous, the two former are used. When sulphur is present, which will be evident from the odour of the oil and its distillates, it is partially removed by treatment with a solution of litharge in strong caustic soda. All these treatments are executed in the experimental stage in glass separators (fig. 25), or in the half-gallon bottles known as "Winchesters." Agitation is accomplished by hand or by attaching the vessel to a shaking machine, of which fig. 26 is an illustration. Miniature agitators are also in use, and are made on the same design as those employed for large quantities.

The acid used is what is known as "strong" (specific gravity 1·840 or 168° Twaddell). The oil to be treated is thoroughly freed from any traces of water (and it will be obvious that distillates from steam distillations and filtrates from brine-cooled chambers are liable to contain such) before the sulphuric acid is added. If this precaution is omitted the operation becomes more costly because the sulphuric acid is diluted; nor is the resultant oil so satisfactorily treated.

The amount of acid required must be determined by experiment. Benzine and kerosene fractions usually require a small percentage only, probably 1 per cent. or 2 per cent., while the heavier oils may take as much as 10 per cent.

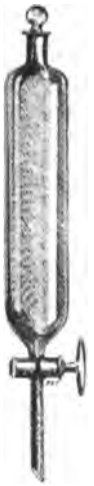


Fig. 25.—Separator (Laboratory).

The acid and oil are agitated together in the mixing vessel, or washer, the duration of the agitation depending on the grade of oil. The mixture is allowed to repose until the tar is precipitated, the period required for the subsidence depending on the "body" of the oil. Benzine takes a short time, kerosene a little longer, while heavy oils take a considerable time to thoroughly separate. When this is complete the oil is divided from the tar, allowing no traces of the latter to be carried over, and transferred to another vessel in which it is neutralised by agitating it with caustic soda solution. The strength of the latter varies with the grade of oil and the amount of acid employed, and varies from 6° Twaddell for benzine to 24° Twaddell for heavier oils. Care must be taken in agitating the acid-treated oil with caustic soda solution, as too violent or too long agitation frequently results in an emulsified mass, difficult and sometimes impossible to separate into oil and solution. While benzine and kerosene will separate from chemicals satisfactorily at ordinary temperatures, steam heat is necessary to effect a separation of the soda liquor from heavy oil. The acid tar, however, usually precipitates, although, as already indicated, somewhat slowly.

Litharge treatment, when necessary, is carried out in similar vessels to those employed for acid and soda. The litharge is dissolved in a strong solution of caustic soda, and a small percentage of the sodium plumbite so obtained agitated with the benzine or kerosene (sodium plumbite has little or no effect on the heavier oils) until the lead sulphide formed begins to separate. This takes the form of a black precipitate, when a fairly large proportion of those sulphur compounds which will combine with the lead salt are present; but, if merely existing in small quantities, a yellow or brown colouration only is produced. The precipitation of this colour may be effected by adding a trace of sulphur to the mixture.

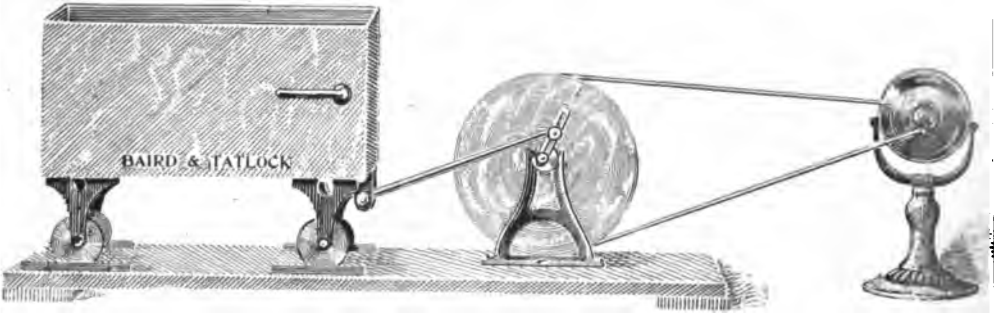


FIG. 26.—Shaking machine.

Sulphur is also partially eliminated by the Edeleanu sulphur dioxide process, which will be referred to later.

Viscosity or "body" of lubricants is the property by which these oils are valued. It is most important to note that the loss of this property when the oil is heated must not be excessive, for an oil which becomes rapidly thin on heating will not be a good lubricant. To discover the extent of this reduction due to rise of temperature the determination of viscosity should be carried out at differing temperatures. In deciding the yields of the various grades obtainable from any crude oil, or in examining the refined products or market samples, the instrument used for determination of viscosity is that designed by Sir Boverton Redwood. Within recent years the viscosity of fuel oils for the requirements of the British Government has been defined within certain limits. The instrument specified for this test in known as the Redwood No. II. viscometer. Both instruments are fully described in the following abstract from the inventor's *Treatise on Petroleum* :—[1]

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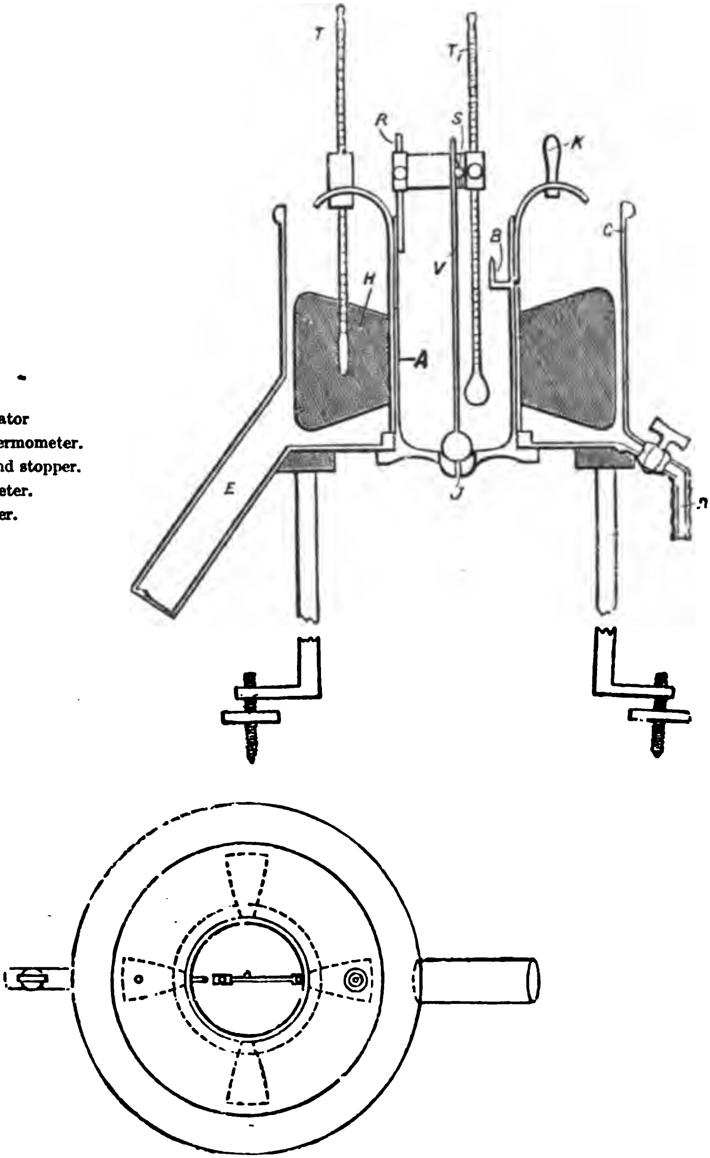
[1] Redwood's *Treatise on Petroleum,* 3rd Ed., vol. ii. pp. 272, 275–277.

"**Lubricating Value.**—The lubricating value of an oil is now usually deduced from its viscosity, though in some cases frictional tests are made with machines which will be described later.

"**Determination of Viscosity.**—Although the results afforded by the use of a viscometer are purely arbitrary, and the viscosity as determined with one form of viscometer is not usually strictly comparable with that ascertained by means of another of different construction, yet each of the viscometers generally employed admits of being accurately standardised, so that any number of instruments of the same form may be constructed to give practically identical results. Viscosity is now regarded as one of the most important characters of a mineral lubricating oil, and several forms of apparatus for determining it are in use.

"The Redwood viscometer has been adopted in this country by the Admiralty, the War Office, the principal Railway Companies, the Scottish Mineral Oil Association, and the petroleum trade generally.

"**Redwood's Viscometer.**—The Redwood viscometer (figs. 27 and 28) is a modification, designed by the author in 1885, of the instrument formerly used at the Battersea Works of Price's Patent Candle Company. It consists of a silvered copper oil-cylinder, 1⅞ inches in diameter and 3½ inches in depth, furnished with an agate jet fitted into a slightly conical metal seating. The cylinder is fixed in a brazed copper water-bath provided with a copper heating-tube projecting downwards at an angle of 45° from the side near the bottom, as well as with a revolving agitator carrying a curved shield to prevent splashing and a thermometer to indicate the temperature of the liquid in the bath. The oil-cylinder has a stopper consisting of a small brass sphere which rests in a hemispherical cavity in the agate jet. The sphere is attached to a wire by means of which it is raised and hooked on to a clip which supports a thermometer in the oil. Inside the oil-cylinder, and at a short distance from the top, is fixed a small bracket terminating in an upturned point, which serves as a gauge of the height to which the cylinder is filled. The instrument is supported on a tripod stand, furnished with levelling screws. Great care is taken in the construction of the agate jets to secure uniformity, and any small differences in the rate of flow are corrected by slightly altering the position of the pointed bracket in the oil-cylinder. The instruments are thus all standardised. The viscometer is used in the following manner :—The bath is filled with a suitable liquid to a height corresponding with the point of the bracket in the oil-cylinder. Water may be used for temperatures up to 200° F., and a heavy mineral oil for higher temperatures. The liquid being at the required temperature, the oil to be tested, which may previously be brought to the same temperature, is poured into the inner cylinder until its level just reaches the point of the gauge. A narrow-necked flask, holding 50 c.c. to a point marked on the neck, is placed beneath the jet, in a vessel containing a liquid of the same temperature as the oil. The ball valve is then raised,



A = Oil cylinder.
B = Bracket-gauge.
C = Bath.
D = Draw-off cock.
B = Heating-tube.
H = Agitator.
J = Agate jet.
K = Handle of agitator
R = Support for thermometer.
S = Hook to suspend stopper.
T = Bath thermometer.
T_1 = Oil thermometer.

Figs. 27 and 28.—Redwood viscometer.

a stop watch at the same time started, and the number of seconds occupied by the outflow of 50 c.c. noted. When oils are being tested at a temperature much above that of the laboratory, a gas-flame is applied to the heating-tube, and the agitator kept in gentle motion throughout the experiment. The maintenance of the exact required temperature of the oil is thus, after a little practice, rendered easy. It is important that the apparatus should stand perfectly level, and that the oil should be free from dirt, water, or other suspended matters, and not have been heated very much above the temperature of the test during the preceding twenty-four hours. For very accurate determinations the apparatus should be enclosed in a glazed cupboard kept at the same temperature as the viscometer when the temperature at which the tests are being made is such as to admit of this being done. In order to express the results in terms of the viscosity of rape oil at 60° F., the standard used with this instrument, the number of seconds occupied by the outflow of 50 c.c. of the oil is multiplied by 100 and divided by 535, the average number of seconds occupied by the outflow of 50 c.c. of rape oil at 60° F. from the apparatus. The result is multiplied by the specific gravity of the oil at the temperature of testing, and divided by 0·915, the specific gravity of refined rape oil at 60° F., a correction being thus made for the difference in the specific gravities of the two oils. The time occupied in the outflow of 50 c.c. of water, at 60° F., is 25·5 seconds.

"The author attaches considerable importance to the employment of agate in the construction of the jet, as this material is not liable to become worn or injured, even if the instrument is subjected to somewhat rough usage. He has had one of these viscometers in almost daily use in his laboratory at temperatures ranging from 70° to 250° F. for the past twenty-five years, and the jet shows no sign of wear. He also regards the provision of an effective stirrer for the bath as highly desirable, for it is essential that the temperature of the oil subjected to the test should be most accurately adjusted and maintained. In the original standardising of the instrument, *slight* departures from the standard size of the orifice may be corrected by placing the gauge-point a little higher or lower. If, however, there be any considerable deviation from the model, especially in respect of the height of the column of oil, the instrument may be standardised to give the same results as the pattern with a certain oil, but will not necessarily furnish concordant results with another oil of different viscosity.

"The author has devised a modified form of his viscometer, which, with the authority of the Admiralty, will be known as the Admiralty type, for testing oil fuel. This instrument, as its name indicates, has been specially constructed for use in determining the viscosity of oil fuel, for contract purposes, and is intended to be employed at a temperature of 32° F. The oil-vessel is of the same dimensions as that of the original instrument, but the agate jet is longer, and of larger bore, the new viscometer being designed to give an outflow in one-tenth of the time occupied in the outflow of a similar volume from the original pattern. The jet is so mounted as to be completely surrounded by the water in the bath. The water-bath, which is of comparatively large size, and is jacketed, has no lateral heating-tube, but is provided with the usual rotating stirrer. The design is registered, and the sole makers are Messrs Baird & Tatlock (London), Ltd. The oil to be tested should be subjected to prolonged cooling at 32° F., by being kept in a refrigerator through the night, or for at least six hours, immediately before being placed in the oil-cup, and should be thoroughly stirred before being transferred. When accurate results are desired, the viscometer should be placed in a refrigerating chamber, the temperature of which is maintained at 32° F."

Colour.—In the examination of products either from experimental or large scale work it is necessary to measure and record the colour of kerosene, lubricants, and wax. Market requirements have dictated that benzine should be colourless.

A reliable and easily manipulated instrument for colour determinations of both solids and liquids is the Lovibond tintometer, which is fully described (by kind permission of The Tintometer Co., Ltd.) below. Wax in course of manufacture is examined in the liquid condition, and this may be readily done by heating the Lovibond cell, filling it with hot wax, and examining it before it begins to solidify. Liquid wax showing 0·74 yellow (or less) in an 18-inch stratum will be practically colourless to the eye when solid.

"**The Tintometer** was designed by Mr J. W. Lovibond, of Salisbury, for his own use in his business as a brewer. The many experiments that were found necessary before success was achieved aroused his interest in the whole subject of colour and colour measurement, so that eventually he fitted up workshops and laboratories, and employed assistants for carrying out original investigation. Several patents were taken out, in almost all civilised countries, and in 1886 the system, as it stands to-day, was practically complete, though the instrument and many details have been improved and protected by recent patents.

"About 1886 Mr Lovibond made public the result of his researches, and from that time to the present has made frequent communications on various branches of the subject to the principal British scientific societies. From the interest taken in these papers arose many queries as to special applications of the instrument and system, and as each definite problem came forward Mr Lovibond and his assistants undertook the necessary investigations, and supplied instruments, in many cases specially designed.

"During the twenty-six years from 1886 to 1912 inclusive, Mr Lovibond has employed from eight to twelve assistants in such work as is here outlined.

"The tintometer has not been 'on the market' during the whole of this time, though a price-list was issued in response to many inquiries. Mr Lovibond has borne the whole cost of the investigation and experimental work. The apparatus is now ripe for application to a great number of trade and scientific purposes; but in any cases where new or further investigation is necessary, Mr Lovibond and his assistants will undertake it at a reasonable charge. The great fact demonstrated by years of tintometer work is that colour is one of the most constant qualities of substances, and that any variation in colour is an index of variation in other qualities. In many cases a colour estimation, or a series of them, gives the quickest and surest index to absolute value for commercial purposes. Where an eye-estimation of colour is taken as an index of value, tintometer estimation will usually be found an absolute index. As the investigators are, necessarily, not experts in all businesses, it is advisable to state questions as clearly as possible, and to send samples of series of materials properly verified for their important qualities, when it is desired to ascertain whether the colour reading can index those qualities.

"The tintometer is an instrument by means of which the depth of colour in liquids and solids can be accurately measured in degrees, placed in their position in a permanent colour scale, and registered for reproduction at any time.

"It consists of a graded series of standards made of coloured glasses numbered according to their depth of colour, and an instrument for holding the glasses and the object to be measured.

"**The Standard Scales.**—Only three colour scales are necessary for investigation work—these are red, yellow, and blue; but for some special purposes, such as for brewers, for the estimation of carbon in steel, etc., scales in other colours are found convenient. Each ordinary scale consists of glass slips all of one colour, but differing in depth, the divisions of difference being equal throughout, forming degrees of units similar in character to the temperature divisions on a thermometer scale, or to the inches on a foot-rule.

"The colour units are not only of equal depth throughout each scale, but have also a colour equivalence in relation to each other; that is, a given number of units in one scale have an equivalence of colour value in relation to the same number of units in each of the other two scales, so that, upon combinations of equal units of any two, or of the three, a colour nomenclature is founded, which consists of eight fundamental terms—namely, six colour terms and two light terms—by means of which every possible colour can be first measured and then described.

"The standard glasses, as already stated, are three: red, yellow, and blue, in even graduation from ·006 to 20 tint units. In practical work ·01 is found to be the lowest unit essential. Below is appended a table showing the graduations of units for each colour, from which it will be seen that there are 155 for each colour, and 465 in all for the three colours. Such a large number as this is rarely required, except for experimental or research work of considerable extent and variety. In the commercial applications of the tintometer, when a new use is suggested by a business man, the necessary investigations will be undertaken by Mr Lovibond or his staff, and the set supplied for that particular business will only include the glasses (usually very few) that are found necessary. If at any time additional standards are required they can be supplied.

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"These standards are converted into colour constants by their having been co-related to some physical colour constants, as described under the heading of 'Checking the Unit' (p. 78). It is this co-relation to a physical constant which gives the power of defining a colour sensation.

"**The Origin of the Unit.**—This rests primarily on the power of selective light absorption resident in coloured glass. When due care is exercised in the manufacture and the grading of the glass, it is found possible to construct from the colours red, yellow, and blue, three separate scales, the divisions of which shall not only be equal throughout the length of each scale, but also a given number of units in one scale shall have an equivalence of colour relation or value with the same number of units in each of the other scales.

"This equivalence of colour value is accomplished when a normal white light can be gradually absorbed to extinction without the development of any colour by successive additions of an equal number of units of the red, yellow, and blue glass. The luminous intensity of a light so absorbed is then represented by the number of units required for this total absorption. The combined absorptive effect of these three scales is called *neutral tint.* Therefore the starting-point of the system is the *neutral tint unit*, which is the quantity of normal white light absorbed by one red, one yellow, and one blue glass standard unit combined.

"**The Orange, Green, and Violet Unit.**—Upon the principle of colour equivalence, any two of the three glass standard colours absorb all the colour rays of normal white light except that of the one situate between them in the spectrum order, in this way :—An equal number of red and yellow glass standard units will absorb their equivalent numbers of all the colour rays except that of the orange, which is the only colour ray they both transmit.

"An equal number of yellow and blue standards absorb all the colour rays except green, which is the only colour they both transmit.

"An equal number of blue and red standards absorb all the colour rays except violet, which is the only colour they both transmit.

"The colour transmitted by equal units of any pair of glass standards is called *normal colour.* Therefore the *normal colours* are orange, green, and violet, and a single unit of orange is the light transmitted by one red and one yellow standard unit.

"A single unit of green is the light transmitted by one yellow and one blue standard unit.

"A single unit of violet is the light transmitted by one red and one blue standard unit.

"**The Red, Yellow, and Blue Unit.**—Each of the above three colours is the light transmitted by the corresponding coloured glass standard. These are called *the glass standard colours.*

"We have now the means of producing any tint of the six spectrum colours—red, orange, yellow, green, blue, and violet; also of producing a neutral tint of any depth, from the faintest neutral grey to black.

"These may be arranged and defined as follows :—

"Neutral tint or black is the light absorbed by equal values of the three standard glass colours—red, yellow, and blue.

"Standard dominant colours—red, yellow, and blue—are respectively the lights transmitted by the corresponding glass standard colour.

"Subordinate colours—orange, green, and violet—are respectively the lights transmitted by equal values in the two necessary glass standard units.

"As it is an experimental fact that every daylight colour sensation is that of one or of two of these measurable colour rays combined in definite proportion, together with the accompanying daylight which brings the colours within the discriminating power of the human vision, it follows that every daylight colour that can be matched to the vision by the standard glasses can be stated in the terms of colour and depth on the standard glasses used.

"**The Light Unit.**—*The Composition.*—The composition of the light unit is a mixture of the six colour rays, *red, orange, yellow, green, blue, and violet,* and of diffused daylight in equal proportions.

"*The Scale.*—The scale consists of the 364081 wave-lengths which lie between A 760400 and H 396319 of the diffraction spectrum.

"*The Unit.*—The unit is one-hundredth part of the whole scale 364081, and therefore contains 3640 wave-lengths.

"Professor Rood divides the spectrum scale of 100 units into five colour divisions of 20 units each.

"Therefore each colour division contains 72812 wave-lengths.
,, ,, ,, unit ,, 3640 ,,

"The tintometrical colour scales and unit divisions are in exact accord with Professor Rood's divisions, the unit readings of the scales are co-relatable into their equivalent wave-length values; the only difference being that the tintometrical scales comprise mixtures of red and violet which are not seen in the continuous spectrum.

"*The Smallest Perceptible Difference.*—A single unit in the colour scales is divided into 100 perceptible differences in the lighter shades; therefore the vision power of differentiation is limited to 36 wave-lengths.

"**The Dimensions of the Unit.**—The intervals between the units, or main divisions of the scales, are the smallest differences between which the normal vision can discriminate in the deeper shades of glass colours; these are subdivided into tenths as the shades get lighter, and ultimately into hundredths in the very light shades; in fact, increasing in minuteness as the discriminating power of the eye increases.

"The actual dimensions of the unit are arbitrary and of no real importance, so long as the divisions are equal both in dimensions and equivalence, and the subdivisions are sufficiently minute for the work required; and in this sense the colour unit of this system is neither more nor less arbitrary than the inch divisions on a foot-rule or the mass of water chosen to represent a gramme weight, or indeed any other standard unit of measurement.

"**Checking the Unit.**—So far as we are able to judge, no arrangement of the spectrum colours of intense lights is practically applicable for checking the minute divisions of these glass colour scales or their combinations.

"But even without the aid of the spectrum we are not deficient in means of checking the original dimensions of the several colour units, as when the colour of a stable freshly-prepared chemical solution of known percentage has been once accurately measured and recorded, that record is always a witness to the validity of the unit, and any departure under similar conditions of examination would be evidence of error in the scales.

"Also any such error could be defined and corrected by means of the freshly-prepared percentage chemical solution; so that if every existing set of colour standards was suddenly destroyed, new scales exactly corresponding to the originals could be prepared from a series of these solutions.

"**The Colour Nomenclature.**—The system of colour nomenclature is based on the fact that under ordinary daylight conditions the vision is only simultaneously sensitive to two of the six colour rays (which, when united, produce normal white light), the colour sensations of the remaining four being lost in what, for the want of a better expression, may be termed the luminous intensity of the accompanying daylight.

"These two colours are always a dominant, with one of the subordinates adjacent to it in the solar spectrum; the violet must be here considered as lying in a circle, between the red and blue.

"Orange must be in combination with either red or yellow, constituting in their various proportions all the red orange and all the yellow orange colours.

"Green must be in combination with either yellow or blue, constituting in their various proportions all the yellow green and all the blue green colours.

"Violet must be in combination with either blue or red, constituting in their various proportions all the blue violet and all the red violet colours.

"The complete list of colour terms in this system, apart from the term light and neutral tint, are as follows :—

Pure Colours.	*Mixed Colours.*
Red	Red Orange
Orange	Yellow ,,
Yellow	Yellow Green
Green	Blue ,,
Blue	Blue Violet
Violet	Red ,,

Neutral Tint or Black
Light

"There are only six possible colour combinations; the constituents are, however, distinguishable in any proportion. The combinations are:—

Red with Orange
Red ,, Violet
Yellow with Orange
Yellow ,, Green
Blue with Green
Blue ,, Violet

"Some colours, such as certain aniline dyes and a few natural colours, are brighter than standards. Before such colours can be measured, they must be dulled to the measuring point by the interposition of neutral-tint glasses. The unit value of these is added to the colour measurements as light units, and forms part of the measurement, as in the case of the physical colour-constant potassium permanganate.

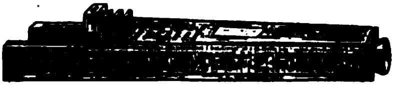

Fig. 29.—Tintometer, monocular form.

"Colours less bright than standards require three glasses to measure them; the least in value of these is a register of the white light absorbed, and forms part of the equation as black units, as in the case of the physical colour-constant cobalt sulphate.

"The names of the colour and light factors in the second half of the equation are the nomenclature by which that particular sensation is known.

"**The Instrument,** which is illustrated by fig. 29, consists essentially of a double tube, ending in an eye-piece at one end, and in equal apertures for viewing the colour to be measured, and the glasses used as measures, at the other end.

"Fig. 29 illustrates the monocular form, now only used for cells of greater depth than 2 inches; for general work an improved form is now preferable. This may be used either as a monocular or as a binocular, and is illustrated in fig. 30.

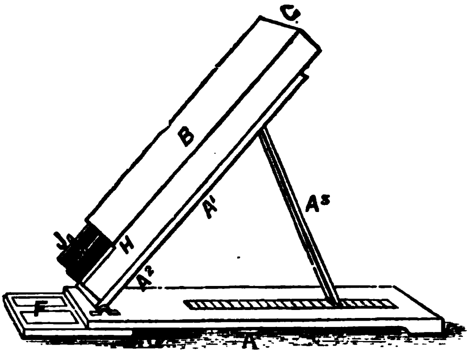

Fig. 30.—Tintometer for opaque objects.

"Fig. 30 shows the arrangement for measuring colour in opaque objects. The optical instrument, B, fits into the shoe of A, the bottom of which is commanded by both tubes of one instrument. Under one side at F is placed the opaque substance to be measured, and under the other the standard white, for reflecting the beam of white light, which is then dissected at J by the suitable standard glasses, as already described for transparent colours.

"Fig. 31 represents the instrument as arranged on the cover of the box, for measuring colour in liquids up to 2 inches in thickness. The instrument, E, slides into the upright stand, D, which fits into the two halves of the cover of the box, B^1 B^2, these being retained in position by the springs, C^1 C^2. If the light is obtained from a high window, the reflector, H, must be fitted into the cover, B, and the box placed so that both the openings are equally illuminated on looking through the eye-piece, G. If the light is obtained from a low window, the reflector, H, is not required, but instead, place a piece of white tissue paper smoothly on the window, and then place the box in such a position that the two openings are equally illuminated on looking through the eye-piece, G.

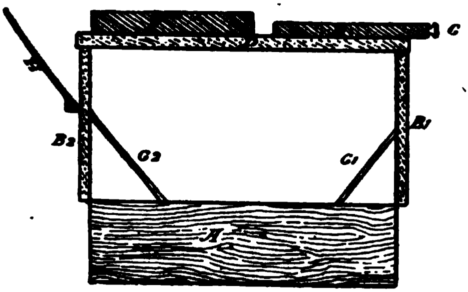

FIG. 31.—Tintometer arrangement for dark-coloured oils.

"A separate stand is required for cells which are longer than 2 inches. The method of arrangement is shown in fig. 32, where one end of the cell rests on the stand, A, which also carries the optical instrument, B, whilst the other is supported by a separate stand, F, which can be moved to accommodate a tube of any length. The reflector, D, is supported on a separate stand.

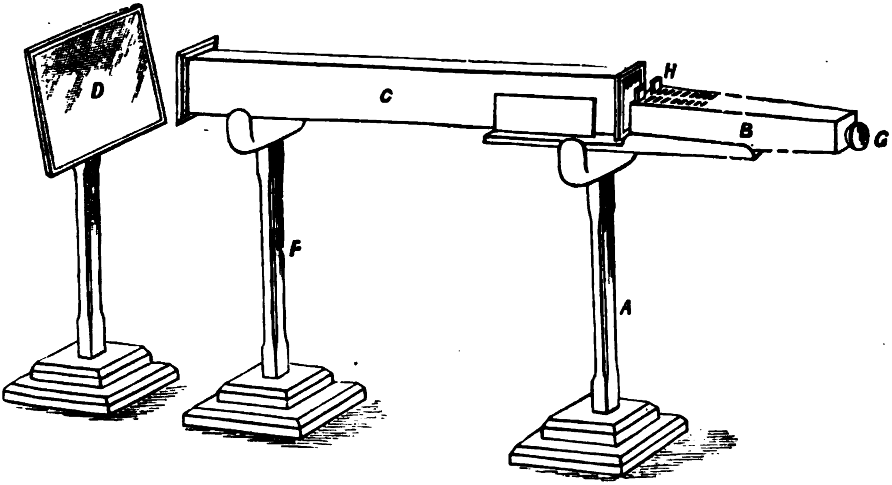

FIG. 32.—Monocular instrument, with long gauged trough and white reflector. For water and similar pale liquids. (Kerosene and paraffin wax.)

"The instrument, in all its forms, is of extreme simplicity, a quality only arrived at after the careful testing of (literally) many hundreds of variations and suggestions made by interested workers and others, with a view to rendering the instrument more universally useful.

"**Instructions for using the Instrument.**—The instrument must be placed facing the light; any ordinary daylight will do, but a north light is preferable. Direct sunlight must not be used. Then adjust the instrument so that equal quantities of light pass up each tube. This is effected by placing a piece of paper, or some evenly-covered surface, or preferably a slip of mirror-glass, under both tubes, and slightly turning the instrument towards the light, until both fields of view are evenly illuminated. When this is effected either side can be used for the standard white without affecting the measurement.

"The coloured light from the object to be measured is transmitted through one tube, and the light from a standard white through the other; this standard white light is then intercepted by the graded coloured glasses until it corresponds in colour with the object to be measured, when the numerical colour value of the glasses used can be read off.

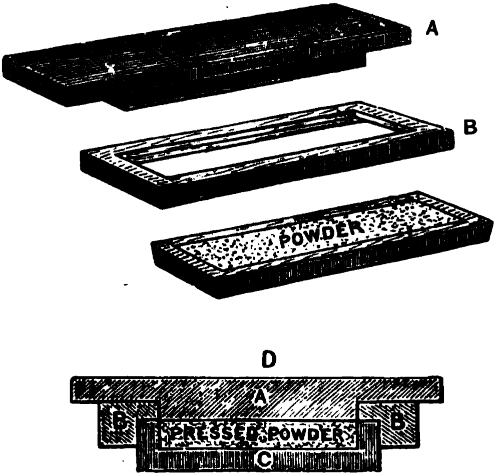

Fig. 33.—Tintometer—powder tray.

"The standard white is pure precipitated lime sulphate, pressed evenly into a tray. In order to ensure uniformity of pressure, the above apparatus is designed for pressing two volumes into one.

"A represents two pieces of glass, one larger than the other, cemented together, the smaller piece just fitting into B, which is a rectangular frame $\frac{1}{10}$th of an inch deep; this fits to a tray, C, also $\frac{1}{10}$th of an inch deep. These are fitted together, then filled with the standard white, and struck evenly off; the powder is pressed lightly down with the small side of the glass presser in order to remove the frame, B, more easily from the tray, C. The broad side of the presser is used to compress the powder even with the sides of the tray. The whole arrangement is shown in section at D.

"If the substance is a yarn, it should be wound evenly on a flat block; if cloth or paper, pinned evenly to a block; if in powder, pressed evenly into one of the special trays.

"When a liquid has to be measured it should be poured into one of the gauged cells, or the capillary film apparatus filled with it.

"**Analysis of colours—Methods of procedure and arrangement of the instrument—Formulating the results.**—When the analysis of any colour is required, the following simple preparations and precautions should be observed:—

"The best way to work is in a room illuminated by north light, and should there be surrounding objects, such as trees, houses, etc., which obstruct in any way the light, it is advisable to utilise pure white tissue paper pasted by the four corners over the whole of that part of the window which illuminates our standard surface.

"Having thus obtained an even, colourless illumination, it is necessary to so arrange the white surface that it is uniformly lighted, that is, that one side is not brighter than the other; this will not be found difficult if the white reflector is placed at an angle with the windows and the tintometer is arranged about the same angle with the standard surface, and then the latter shifted till the rectangular openings are seen to be equal in illumination.

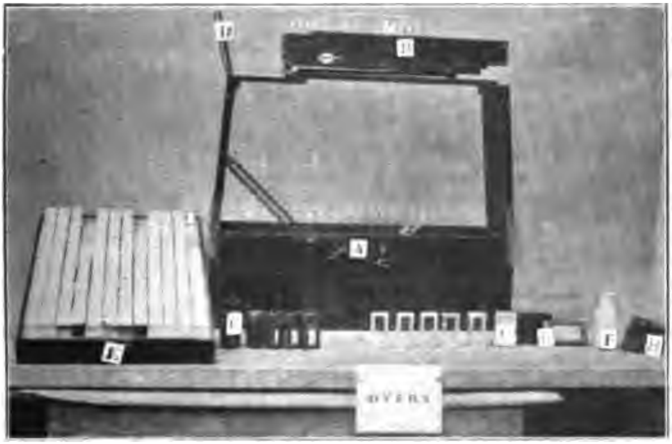

FIG. 34.—Tintometer—instrument, stand, colour glasses, cells, etc.

"The adaptability of the eye to perceive variations in colour varies considerably with time of observation. It is imperative, therefore, to limit the observation to a fixed time, for which five seconds will be found convenient.

"The above directions should be carefully read and implicitly followed, and then accurate readings will be obtained.

"In the following examples of colour analyses, those which are sufficiently stable for physical colour constants are marked as having been co-related to the corresponding unit values in the table of standard glasses (p. 76), and are therefore available for checking the validity of the unit divisions of the standard glasses.

"EXAMPLES OF COLOUR ANALYSES.

Distilled Water (Physical Colour Constant).

	Standard glasses used.					Colour sensation transmitted.		
	Red.	Yellow.		Blue.		Green.		Blue.
2 feet must be matched by	..	·19	+	·34	=	·19	+	·15
4 ,, ,, ,,	..	1·0	+	1·45	=	1·0	+	·45

Tannin Extract—50 per cent. Solution.

	Red.		Yellow.		Blue.		Black.		Orange.		Yellow.
In 1-cm. stratum	3·2	+	15·2	+	·32	=	·32	+	2·88	+	11·0

Glycerine.

	Red.		Yellow.		Orange.		Yellow.
In 5-inch stratum . .	0·20	+	0·32	=	0·20	+	0·12

Cotton Seed Oil (Perine Summer Yellow).

	Red.		Yellow.		Orange.		Yellow.
In 5¼-inch stratum . .	7·1	+	35·0	=	7·1	+	27·9"

For a complete study of this subject reference should be made to Mr Lovibond's latest book.[1]

The quality of the kerosene produced is an important consideration, and, besides the determination of specific gravity, flashing-point, and colour in the manner already explained, it is usually necessary to assess the value of the oil as an illuminant, its rate of consumption, whether it "smokes" or not when burned in an ordinary lamp, and whether it deposits a "film" on the glass chimney of the lamp.

In Sir Boverton Redwood's *Treatise on Petroleum*, vol. ii., 1913, will be found descriptions of several methods of determining the illuminating power of kerosenes. The only practical method, however, is to burn the oil in the lamps sold in the market in which the kerosene is to be placed and to compare its behaviour through a six hours' test with oils competing in the same market. The consumption may be checked by weighing the lamps before and after the test.

"Filming," a white deposit which forms on the glass chimney, is found when kerosene containing appreciable quantities of sulphur

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[1] *Light and Colour Theories*, J. W. Lovibond (E. & F. N. Spon, Ltd.), 1915.

compounds is burned. Results of experiments have shown a marked reduction in this defect by the removal of all but traces of the sulphurous bodies. When certain oils are burned they are very easily affected by slight irregularities in the supply of air, and "smoking" of chimneys, even when the flame is maintained at normal height, results.

Dr Edeleanu, the eminent Roumanian chemist, found that this "smokiness" could be removed from kerosene by treating it with sulphurous dioxide, and perfected a process and plant with that object in view. Installations erected at Rouen and Ploesti were successfully worked on the manufacturing scale, which will be described later. This treatment also reduces sulphur-containing bodies to the minimum; consequently, the process is beneficial for oils suffering from the two defects mentioned above.

The Testing of Pitch[1] involves a considerable amount of work, as will be seen from the following section.

The Testing of Pitch or Asphalt.—In testing a sample of asphalt the following points are usually noted :—

(1) Specific gravity at 25° C.
(2) Appearance—
    (*a*) Colour.
    (*b*) Fracture.
    (*c*) Hardness.
    (*d*) Odour.
(3) Softening point.
(4) Flow point.
(5) Ductility.
(6) Penetration.
(7) Loss on heating.
(8) Penetration after heating.
(9) Bitumen soluble in carbon bisulphide.
(10) Malthenes = bitumen soluble in petroleum spirit.
(11) Carbenes = bitumen soluble in carbon tetrachloride.
(12) Fixed carbon.
(13) Ash.

SPECIFIC GRAVITY.

The specific gravity of asphalt is taken at a standard temperature of 25° C. This temperature has been chosen as a more convenient working temperature for average conditions than 15° C.

The method of determination is as follows :—

A ½-inch test-tube is taken and cut off about 2 inches from the

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[1] *The Modern Asphalt Pavement*, Clifford Richardson.

bottom, the edge is then flared out so that it can be suspended for weighing purposes by a fine wire.

This tube is then weighed in air and in water at 25° C.

About 10 grammes of the asphalt to be tested are poured in the molten state into the tube, which is then suspended in an oven at a temperature just sufficient to keep the asphalt molten.

This serves to drive off any moisture or contained air-bubbles.

The sample is then cooled and weighed accurately both in air and water at 25° C.

Taking

Weight of tube in air $=a$
,, ,, and sample in air $=b$
,, ,, in water $=c$
,, ,, and sample in water $=d$

then—

$$\frac{b-a}{(b-a)-(d-c)}=\text{specific gravity.}$$

Softening and Flowing Points.—Homogeneous mixtures of several non-crystalline hydrocarbons, such as asphalts, can have no definite melting-point.

It is therefore necessary to determine the points at which they lose form, or the softening point, and the point at which they flow.

The methods employed are quite arbitrary, and the results obtained vary slightly with these methods.

(1) A glass crystallising dish, 2 inches to 2½ inches diameter and 1½ inches deep, is filled with mercury to within ⅝ths of its capacity and placed on a wire gauze over a small flame well protected from draughts.

The specimen to be tested is placed on a No. 2 microscope covering glass floated on the mercury.

In the case of a hard asphalt several meal-like grains are placed on the slide, and in the case of a soft asphalt it is drawn out to about 1 mm. thickness and then several 1-mm. lengths cut off and placed on the glass. The dish is now covered with an inverted funnel with a stem of sufficiently large diameter to allow a thermometer to be passed through, which is held in position by a clamp.

The flame is regulated so that the rate of heating is from 3° to 5° per minute.

The point at which the grains begin to lose form is taken as the "softening" point, and that at which they flow and form a sheet on the glass is taken as the "flow" point.

(2) Cover the bulb of a thermometer with the asphalt to be tested, immerse in a beaker of water, and heat gradually with constant stirrings.

The temperature at which the asphalt begins to lose form is the "softening" point, and that at which it begins to flow up the thermometer is the "flow" point.

(3) **Kovac's Method.**—This method is very easy to work and gives very concordant results. The apparatus (fig. 35) consists of a copper or brass plate $\frac{1}{8}$ inch thick, $3\frac{1}{4}$ inches diameter, fitted with four cups and a thermometer hole, supported on three legs $2\frac{3}{8}$ inches high. These cups have the following dimensions, with an aperture at the bottom :—cups, inner diameter, $\frac{1}{2}$ inch ; height, $\frac{5}{8}$ inch ; aperture, $\frac{3}{32}$ inch. The cups are filled with the asphalt to be tested, the thermometer fitted in the hole provided and placed in a beaker 6 inches high by $3\frac{1}{2}$ inches diameter, which is in turn placed in another beaker, $4\frac{1}{3}$ inches diameter, containing rape oil or glycerine, and the whole covered by an asbestos pad.

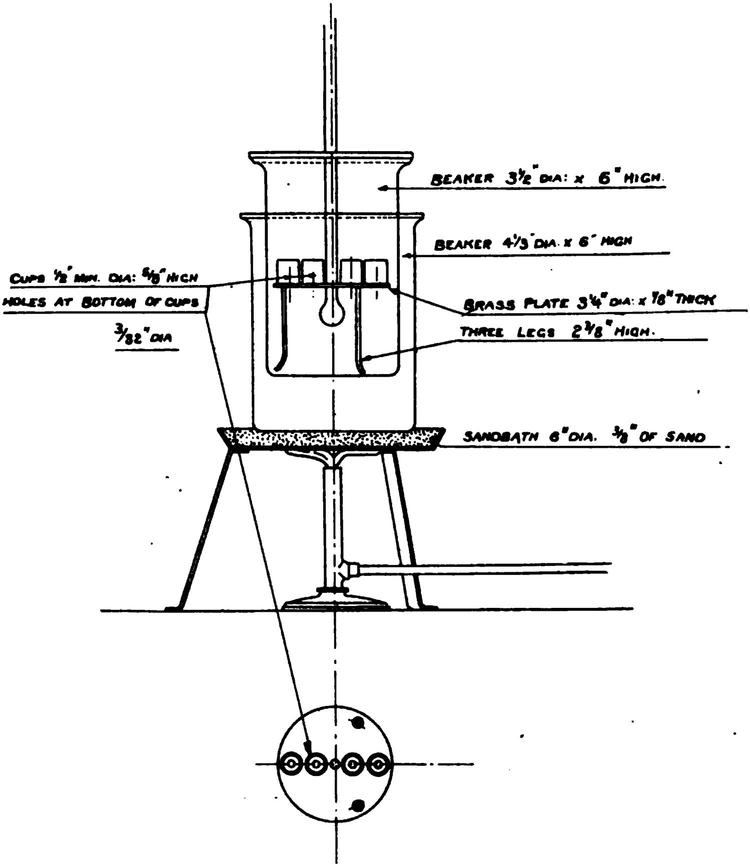

BEAKER 3½" DIA: x 6" HIGH.
BEAKER 4⅓" DIA. x 6" HIGH
CUPS ½" MIN. DIA: ⅝" HIGH
HOLES AT BOTTOM OF CUPS
3/32" DIA
BRASS PLATE 3¼" DIA: x ⅛" THICK
THREE LEGS 2⅜" HIGH.
SANDBATH 6" DIA. ⅜" OF SAND

FIG. 35.—Apparatus for Kovac's method.

The apparatus is now slowly heated, and the temperature at which the asphalt just shows at the bottom of the cups is taken as the "softening" point, while that at which the first drop falls is taken as the "flow" point.

Ductility.—The ductility of an asphalt is a very important property, owing to it having a great bearing on the binding power. It is affected to a large extent by the presence of free carbon, which lowers it according to the amount present. Generally speaking, the greater the ductility of an asphalt after fluxing to a standard hardness the better the quality.

To estimate the ductility of an asphalt a definite hardness is taken as the standard for all tests, and asphalts which have a higher degree of hardness than this standard are reduced to it by fluxing with a heavy residuum oil. The United States standard of hardness is such that when the asphalt is tested with a Dow penetration apparatus it shall give a penetration of 50° at 77° F., using a No. 2 needle with a total weight of 100 grammes acting for 5 seconds. The sample after fluxing to the required hardness is moulded into a standard size block in a special mould, of which part acts as a holder during the test. The test is carried out in Smith's ductility apparatus, which is designed for elongating this block at a definite rate (5 cm. per minute) under water at 77° F. (fig. 36). By drawing out under water in this way the thread of asphalt is supported during the test and does not sag and cling to the base of the apparatus.

The elongation is continued until the thread of asphalt breaks or to a maximum of 100 cm.

The apparatus is very easy to work, the rate of drawing out being regulated by the beats of a metronome.

Penetration.—The penetration of an asphalt is a measure of its softness. It is estimated by allowing a standard needle carrying a definite weight to act for a definite time on a sample kept at a certain standard temperature.

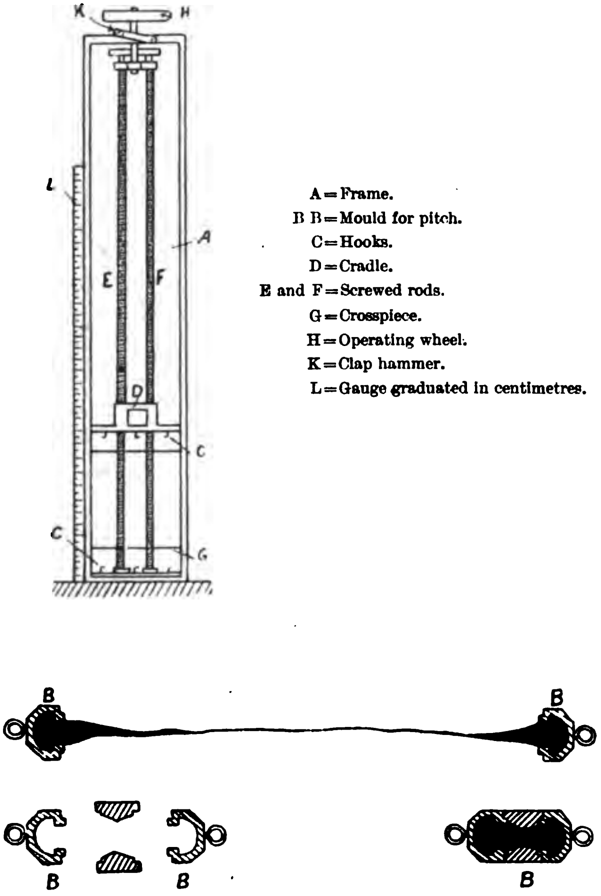
The standard needle is a No. 2 sharps, and the usual conditions of time, temperature, and acting weights employed are as follows :—

(1) At 32° F. with 200 grammes total weight acting for 1 minute.
(2) At 77° F. ,, 100 ,, ,, ,, ,, 5 seconds.
(3) At 100° F. ,, 50 ,, ,, ,, ,, 5 ,, .

By carrying out these tests under the above varying conditions very useful information is obtained as to the behaviour of asphalt with change of temperature. For general purposes, however, the test No. (2) is usually sufficient.

The tests are carried out using a specially designed apparatus (fig. 37), that of Mr A. W. Dow being the best known and accepted as a standard. According to him the method of procedure is as follows :—

"The apparatus consists of a No. 2 needle, A, inserted in a short brass rod which is held in the aluminium rod, C, by the binding screw, B. The aluminium rod is secured in a framework so weighted and balanced that when it is supported on the point of the needle, A, the framework and rod will stand in an upright position, allowing the needle to penetrate perpendicularly without the aid of a support, thus doing away with any friction.

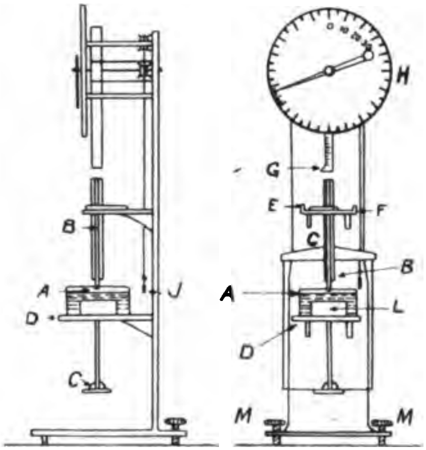


A = Frame.
B B = Mould for pitch.
C = Hooks.
D = Cradle.
E and F = Screwed rods.
G = Crosspiece.
H = Operating wheel.
K = Clap hammer.
L = Gauge graduated in centimetres.

Fig. 36.—Ductilometer (Smith's).

"The frame, aluminium rod, and needle weigh 100 grammes with the weight on bottom of frame ; without weight, 50 grammes. Thus, when the point of the needle rests on the surface of the sample of material to be tested as to the penetration, it will penetrate into the sample under a weight of 100 grammes or 50 grammes as desired.

"The needle and weighted frame are shown in fig. 37, side and front views of the entire apparatus put together and ready for making a penetration. D is the shelf for the sample, E is the clamp to hold the aluminium rod until it is desired to make a test, F is a button which when pressed opens a clamp, E. By turning this button while the clamp is being held open it will lock and keep the clamp from closing until unlocked. The device to measure the distance penetrated by the needle consists of a rack, the foot of which is G. The movement of this rack up or down turns a pinion to which is attached the hand which indicates on dial, H, the distance moved by the rack. One division of the dial corresponds to a movement of the rack of $\frac{1}{100}$ cm. The rack can be raised or lowered by moving counterweight, J, up or down. L is a tin box containing sample to be tested, which is covered with water in the glass cup, thus keeping its temperature constant. M M are levelling screws. A clock movement having a 10-inch pendulum is attached to the wall to one side of the machine. Make a mark, P, on the wall just at the extremity of the swing of the pendulum ; a double swing of this pendulum, that is from the time it leaves P until it returns, is one second.



A = No. 2 needle.
B = Binding screw.
C = Aluminium rod.
D = Shelf.
E = Clamp to hold aluminium rod.
F = Button to open clamp.
G = Measuring device.
H = Dial.
J = Counterweight.
L = Box for material to be tested.
M = Levelling screws.

FIG. 37.—Dow penetration machine.

"The only other things necessary to complete the outfit are a large dish-pan, a pitcher to hold ice-water, and a tin for hot water ; a coffee-pot is a good thing.

"To make penetration tests, place the materials contained in circular tins, along with the glass dish, under 5 or 6 inches of water in the dish-pan, which should have been previously brought to a temperature of 77° F. by the addition of hot water or cold water.

"While the samples are under the water it should be stirred every few minutes, with the thermometer and the temperature kept constant at 77° F. by the addition of hot or cold water as the case may require. The samples should remain under the water for at least fifteen minutes, and in cases where they are very cold or hot, at least one half-hour. The most expeditious way to proceed in testing a sample just taken from a still or tank is to immerse it in ice-water as soon as it has hardened sufficiently and keep it there for ten minutes, then in the water at 77° F. and keep it there for fifteen minutes. When the sample has remained in the water for the specified time it is ready to penetrate.

"The aluminium rod, C, should be pressed up through the clamp, E, so that it will be at such a height that the glass cup will easily pass under it when placed on shelf, D.

"A sample tin box should now be placed in the glass cup and removed in it covered with as much water as convenient without spilling.

"The glass cup containing sample is placed on shelf, D, under C. Insert brass rod with needle into C, and secure by tightening binding screw, B; lower C until the point of the needle very nearly touches surface of sample; then, by grasping the frame with two hands, cautiously pull down until needle is just in contact with surface of sample.

"This can best be seen by having a light so situated that, looking through the sides of the glass cup, the needle will be reflected in the surface of the sample. After thus setting the needle, raise counterweight, J, slowly until the foot of the rack, G, rests on the head of rod, C; note reading of dial, and press button, F, thus opening the clamp.

"Hold open for five seconds, and then allow it to close. The difference between the former reading of the dial and the present is the distance penetrated by the needle, or the penetration of the sample. Raise rack, loosen binding screw, B, raise rod through clamp, leaving the needle sticking in sample. Remove needle from sample, clean well by passing through a dry cloth, replace needle in C, and the machine is ready for another test.

"Do not clean needle on oily cloth or waste.

"Do not allow rack to descend too rapidly on rod, C, as it may force C through the clamp, thus spoiling the reading.

"After using the machine, leave it so that the top of the rack is just level with its base. You will thus prevent dust from entering and getting into pinion. When not in use keep machine covered with a cloth to protect from dust.

"Examine point of needle from time to time with magnifying-glass to see that it is not injured in any way.

"If the needle is found defective, remove by heating the brass rod, when the needle can be withdrawn with pincers. Break eye from one of the extra needles and press into brass rod previously heated.

"If needle does not stay in well, insert it with small lump of asphalt.

"If when this framework is supported on the point of the needle it does not balance so that the aluminium rod, C, stands perfectly perpendicular, the frame is bent, and should be straightened until the rod stands perpendicular. This can easily be done by hand.

"If rack, G, does not descend readily of its own weight when counterweight, J, is raised, it is likely that dust has got into the pinion. To get at the pinion to clean, remove dial, H, and bearing, when pinion can be pulled out sufficiently far to clean.

"Never oil rack and pinion, as it prevents a free movement of rack."

Loss on Heating.—This is determined by heating 20 grammes of the pitch for five hours at 325° F. in a crystallising dish $2\frac{1}{4}$ inches diameter by $1\frac{5}{16}$ inches high, or in a 3-oz. seamless sample tin as used in penetration test.

The exact dimensions mentioned above need not be strictly adhered to, provided they are approximately correct.

Of much greater importance is the maintenance of the correct temperature in the oven, and this point should be carefully watched.

Care also must be taken that the sample box, or dish, does not rest directly on the metal shelf of the oven; it should be rested on a pad of asbestos.

The loss on heating should not be so high as to materially affect the penetration of the sample before and after heating.

DETERMINATION OF BITUMEN CONTENT.

(*a*) Total bitumen.

(*b*) Malthenes or naphtha soluble bitumen.

(*c*) Carbenes.

The method of the determination of these constituents is the same in each case.

The total bitumen is estimated by the solubility of the asphalt in carbon bisulphide, the malthenes by solubility in petroleum spirit of a definite specific gravity, and the carbenes by solubility in carbon tetrachloride.

The determination is carried out as follows :—

One gramme of finely divided asphalt, which should be powdered if possible, is placed in a 200-c.c. Erlenmeyer flask, and covered with 100 c.c. of the solvent to be used. The flask is now corked up and allowed to stand overnight. In the meantime the filtering crucible has been prepared. This crucible is of the Gooch pattern, but of special form, with a large filtering area, the size being as follows :—

Diameter at top, 4·4 cm., at bottom, 3·6 cm.; depth, 2·6 cm. The bottom is covered with a pad of ignited asbestos for filtering purposes, and the whole weighed after ignition. The crucible is now fitted into a filtering flask, and the supernatant liquid poured through, using at the same time a gentle suction. More of the solvent is added to the residue in the flask, and this again decanted through the filter, the operations being continued until the solvent used is clear. Any residue remaining in the flask is now washed into the filter, well washed with the solvent, and then the filter and residue are dried at 100° C.

The difference between increased weight in crucible and original amount of asphalt taken equals bitumen soluble in solvent taken.

The solvent is now evaporated from the dissolved bitumen, which is then ignited, and the weight of any mineral residue found is deducted from the loss of weight originally obtained.

In a good asphalt the bitumen soluble in carbon bisulphide and carbon tetrachloride approximate very closely, but in the case of a badly prepared asphalt, where there has been inefficient distillation, the carbenes are much lowered. The naphtha soluble bitumen is quite an arbitrary quantity. It varies to a large extent with the petroleum spirit employed, the spirits of higher boiling-point being better solvents than those of lower boiling-points.

The solubility also varies with spirits of different origin even of the same boiling-points.

In the United States this determination is carried out using spirit of 88° Baumé (specific gravity ·642) or 62° Baumé (specific gravity ·729). In a temperate climate it is not very easy to work with a spirit of specific gravity ·642, but in a tropical climate it would be impossible; for this purpose, therefore, the spirit of specific gravity ·729 is necessary, and this will only give concordant results when the composition is kept constant.

In any case the results are only comparative.

Mineral Matter or Ash.—This is determined by weighing out 1 gramme of the asphalt in a No. 0 porcelain crucible or a platinum crucible, and igniting until the residue is free from carbonaceous matter. This determination is not quite exact, as sulphuric acid or alkalis may be volatilised, but is sufficiently accurate for technical purposes.

Fixed Carbon.—The fixed carbon content is determined according to the method adopted by the Committee on "Coal Analysis" of the American Chemical Society.

It was published in the *Journal* of this Society, 1899, xxi. 1116. One gramme of the sample to be tested is weighed into a platinum crucible weighing 20–30 grammes, provided with a tightly-fitting cover.

Heat over the full flame of a Bunsen burner for seven minutes.

The crucible should be supported on a platinum triangle with the bottom 6–8 cm. above the top of a ½-inch burner, the flame of which when burning free should be fully 20 cm. high, and the determination should be carried out in a room free from draughts. The upper surface of the cover of the crucible should burn clear, but the under surface should remain covered with carbon.

The residue, minus the ash in the bitumen, is the fixed carbon, and is calculated to 100 per cent. with the volatile hydrocarbons, excluding inorganic matter.

REPORT ON SAMPLE OF PITCH OR ASPHALT.

SPECIFIC GRAVITY at 25° C.

APPEARANCE—
- (*a*) Colour.
- (*b*) Fracture.
- (*c*) Hardness.
- (*d*) Odour.

SOFTENING POINT.

FLOW POINT.

DUCTILITY (fluxed to 50°; penetration at 77° F.) at 77° F.

PENETRATION—
- (*a*) At 32° F., No. 2 needle, with 200 grammes total weight, 1 minute.
- (*b*) At 77° F., No. 2 needle, with 100 grammes total weight, 5 seconds.
- (*c*) At 100° F., No. 2 needle, with 50 grammes total weight, 5 seconds.

LOSS ON HEATING (dry substance)—5 hours at 325° F.

PENETRATION AFTER HEATING—
- (*a*) At 32° F., No. 2 needle, with 200 grammes total weight, 1 minute.
- (*b*) At 77° F., No. 2 needle, with 100 grammes total weight, 5 seconds.
- (*c*) At 100° F., No. 2 needle, with 50 grammes total weight, 5 seconds.

TOTAL BITUMEN soluble in carbon bisulphide, air temperature, mineral ash.

SPECIFIC GRAVITY, ·830. SETTING-POINT, 76° F.

13	14	15	16	17	18	19	20
53	·855	·864	·869	·875	·878	·896	
ter-	..	..	..	..	..	..	Residue, coke, and loss.
diate	..	..	..	..	..	..	
il.	..	..	..	..	..	..	
1	..	..	..	..	..	..	
75	..	..	..	..	..	..	
27	249	277	320	348	380	325	
48	52	63	86	101	114	125	

Heavy oil and paraffin.
Each fraction cooled to 32° F. and pressed.
Scale re-pressed.

.	0·2	0·6	1·4	2·1	2·4	2·0
.	112	114	116	122	132	140
.	4·8	4·4	3·6	2·9	2·6	3·0
.	·863	·878	·902	·928	·937	·942
lwood,	35″	37″	44″	72″	125″	300″

ted.

i

MALTHENES.—Bitumen soluble in naphtha, specific gravity ·729. This is per cent. of total bitumen.

CARBENES.—Bitumen soluble in carbon tetrachloride.

FIXED CARBON.—Bitumen yields on ignition.

MINERAL ASH.—Bitumen yields on ignition.

VOLATILE HYDROCARBONS.—By difference.

Conclusion.—The foregoing pages have suggested the variety of crude oil refining possibilities contingent on the quality of the crude oil and the exigencies of the market, and have described methods and instruments customarily employed for that detailed analytical examination of the crude to be dealt with, which must be the preliminary step to any decision as to the nature and scope of refining operations to be embarked on, on a commercial scale. The data thus secured, considered in the light of marketing conditions, will enable that decision to be wisely made.

Diagram 2 illustrates a form of report from which results may be seen at a glance, and Diagram 3 the "flow" of the oil through a complete plant.

The quantities to be dealt with will be approximated very closely from the percentages shown on Diagram 2, and the necessary plant specified to suit each stage of the process.

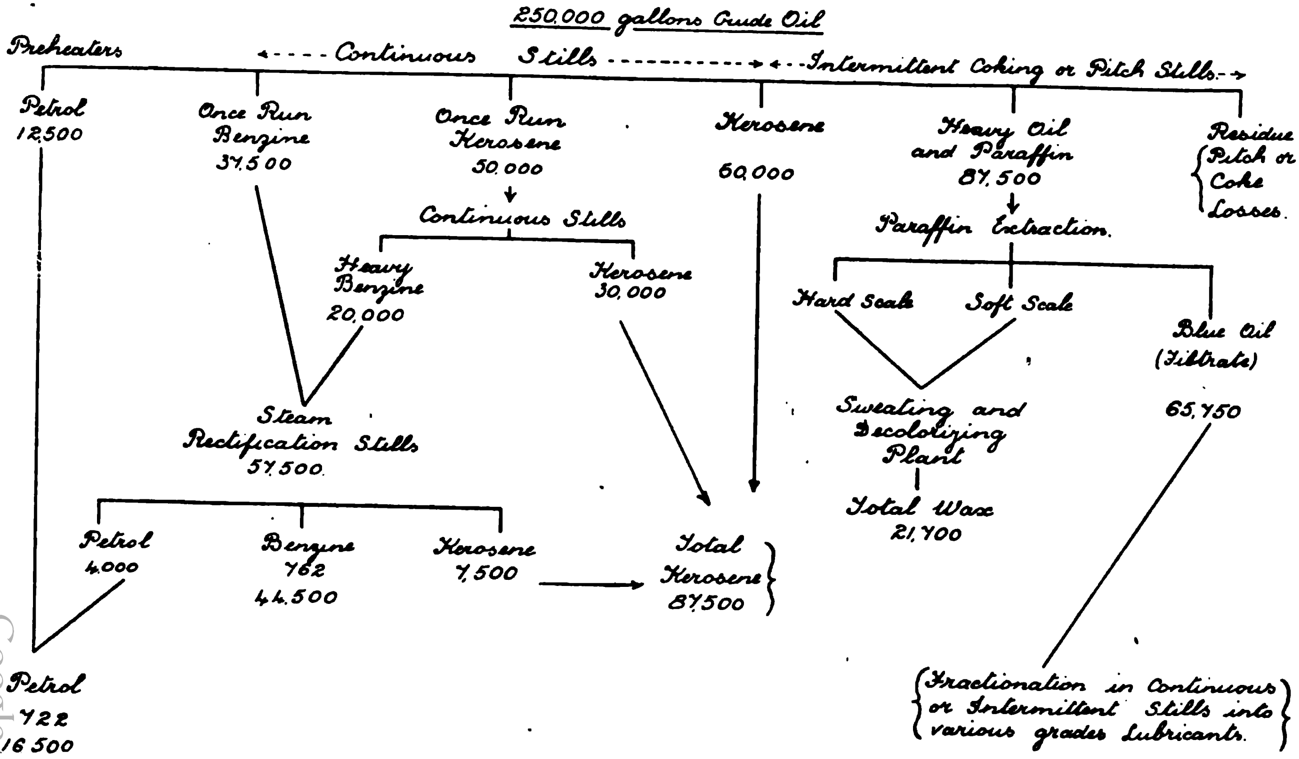
In the actual operation of refining it is imperative that crude oil supplies should be examined periodically, and the results compared with that obtained from the large quantities. Whether a full or only partial examination is necessary will depend on the variation in the composition of the material.

The next step is the selection of plant to carry out the process decided upon, and also of a site on which to lay it out. This must be considered in conjunction with suitability of the land for building, facilities for transport, possible water supply, and drainage outlet.

Full refining operations may be divided into and considered under the following headings :—

1. Departments general to the whole establishment (Chapter II.).
2. Storage of crude oil and products (Chapter III.).
3. Distillations (Chapter IV.).
4. Paraffin extraction and refining (Chapter V.).
5. Candle manufacture (Chapter VI.).
6. Chemical treatments (Chapter VII.).
7. Distribution of products (Chapter VIII.).
8. Engineering specifications (Chapter IX.).

DIAGRAM 3.


250,000 gallons Crude Oil
Preheaters
←- - - - Continuous Stills - - - - - - - - - - - →←- - Intermittent Coking or Pitch Stills - →
Petrol 12,500
Once Run Benzine 37,500
Once Run Kerosene 50,000
Kerosene 60,000
Heavy Oil and Paraffin 87,500
Residue {Pitch or Coke Losses.
Continuous Stills
Heavy Benzine 20,000
Kerosene 30,000
Paraffin Extraction.
Hard Scale
Soft Scale
Blue Oil (Filtrate)
65,750
Steam Rectification Stills 57,500
Sweating and Decolorizing Plant
Total Wax 21,700
Petrol 4,000
Benzine 762 44,500
Kerosene 7,500
Total Kerosene 87,500
Petrol 722 16,500
{Fractionation in Continuous or Intermittent Stills into various grades Lubricants.}

Showing process for working 250,000 gallons daily of crude petroleum of composition shown on Diagram 2.
(Quantities in round numbers in gallons. Chemical treatments unnecessary in this instance.)

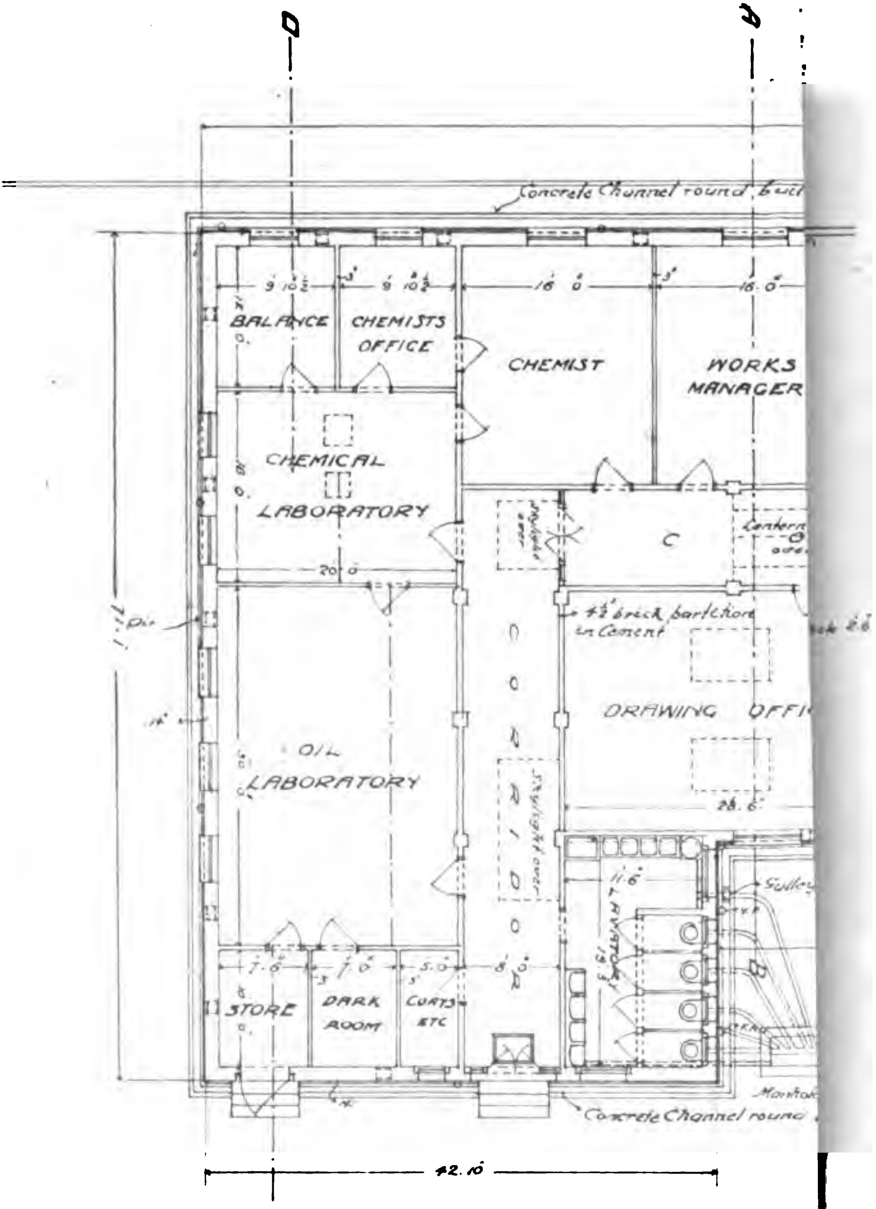

D
A
Concrete Channel round Buil
9 10½
9 10½
16 0
16 0
BALANCE
CHEMISTS OFFICE
CHEMIST
WORKS MANAGER
CHEMICAL LABORATORY
20 0
C
Skylight over
4½ brick partitions in Cement
DRAWING OFFI
28.6
OIL LABORATORY
CORRIDOR
Skylight over
11.6
LAVATORY
19.3
Gulley
7.6
7.0
5.0
8.0
STORE
DARK ROOM
CURTS ETC
Manhole
Concrete Channel round
42.10

FIG. 38

CHAPTER II.

DEPARTMENTS GENERAL TO THE WHOLE REFINERY ESTABLISHMENT.

It is well that these should be considered at the outset, so that prospective refiners of petroleum may realise what are necessary for the formation of a complete refinery. The designer will, of course, determine the dimensions of each section to suit the quantity of crude oil to be dealt with. It is assumed that a site has been selected with good facilities for sea, railway, and road transport; that the ground is suitable for building, without undue expense in foundation work; that ample water is available, and no difficulties exist regarding drainage outlet. Wherever a refinery is erected the rules laid down by Government or local authorities must be adhered to. Strict conformity thereto in the beginning may prevent trouble and expense later on.

Whether coal, gas, or oil is to be employed as fuel must be decided before operations can go very far, as each requires special arrangements for consumption. If the refinery is situated near the oil-fields, oil will be selected, or, possibly, natural gas; but if overseas, whether oil, coal, or producer gas is selected is a matter to be settled by experienced engineers, and is simply a question of cost.

Offices and Laboratory.—Fig. 38 shows a very convenient plan of an office arranged to the best advantage for centralisation of control of all departments. The laboratory is intended for routine testing and examination of crude materials, products, stores, etc., and not for scientific research, which, in the writer's opinion, is best conducted away from the works influence. It will be seen that all departments are provided for in the arrangement drawn, and all should be connected to the various sections of the refining plant through a telephone exchange.

Workshops and Stores Department.—The design and equipment of these will depend on the situation of the refinery. If in a position where supplies are difficult to obtain and no facilities for repair work exist, it will be necessary to instal these departments sufficiently large to cope with all the requirements of the plant for both repairs and stores. It need scarcely be said that all workshop appliances must be of the latest labour-saving designs, and the stores arranged on the best lines for the issue of demands with a minimum of labour. The office work must necessarily be superintended by a man having experience in time-keeping, pay, costs, general accounting, and correspondence. The preparation of a good cost system is imperative for each section of the process, the whole being embodied in a summary, and will best be undertaken by an experienced accountant acquainted thoroughly with the process of manufacture.

Electric Lighting.—The plant should be well lit throughout by the most economical lamps, arranged to the best advantage having regard to the position of the various departments, tail-houses, worm ends, paraffin extraction sheds, and refining plant, in fact, in every place where work is carried on at night time. Electric hand-lamps should be used when it is necessary to work in positions at some distance from the light or in the shadow—tank dipping, for instance.

Steam Supply.—A large amount of steam is required for the distillation of petroleum, and may be supplied from a central battery of boilers or from smaller units attached to each section of the plant. The former is the better arrangement, and where a large and complete refinery is being laid out, may be placed in an advantageous position. In some refineries, built originally on a small scale and extended to a large capacity during a period of years, the latter was found the only convenient means of steam distribution. The Lancashire type of steam boiler has been largely employed in refinery practice, but many other types are equally efficient and in some cases more cheaply and quickly erected. The selection of the type most suitable may well be left to the experts responsible for the design of the plant. If oil is to be used as fuel, the steam supply will be largely increased, over the amount required for distillation, if the oil is atomised in a "burner" or injector by steam pressure on entering by the furnace of either steam boiler or still. There are several types of oil burners, and full particulars of these will be found in numerous books of reference on the subject of liquid fuel (see Appendix).

The chief systems are oil forced through the "burner" by (1) pressure, the construction of the burner atomising oil; (2) by atomising the oil passing through the burner with a current of compressed air; and (3) employing steam in place of air. Because of the greater ease with which the latter may be worked by firemen it is the system almost universally used in refineries where oil is burned as fuel.

Power.—In the modern refinery steam engines for pumping and machinery (paraffin extraction, treatments, etc.) have been displaced by electrical power generated in a central station.

Traction and Roads.—If coal is to be used as fuel, railways must be laid for carrying it to the various points of consumption and arrangements made for the disposal of ashes. A locomotive (steam, oil-fired) may be found the most suitable means of traction in this case.

Roads must be made for communication between the various sections of the plant. It will be obvious, therefore, that in designing a refinery for the process of the full refining of petroleum, the layout of roads and railways will entail careful consideration in conjunction with local conditions, which will determine the class of work to be undertaken in this connection.

Water Supply and Drainage.—These may be considered conjointly. Water is required for steam raising, condensation, and chemical treatments, as well as a supply of potable water for the use of workmen.

It is best to employ fresh water for condensation, although brackish or even salt water has been and is employed for this purpose. In some cases, where the water contains large quantities of matter in suspension, settling ponds are employed in which the water is allowed to repose until the silt subsides. The cleaning of the ponds recurs periodically, and adds to the expense of the installation.

Fresh water reduces to a minimum the repairs to plant necessitated by the corrosive action of brackish or salt water.

For steam and chemical treatments, good fresh water is necessary. The situation of the refinery may be such that a reservoir to contain several months' supply will be necessary. This is the case in tropical countries, where the dry season is prolonged, the only fresh water available being rain which falls during certain periods of the year. However, the situation may be more favourable, and fresh water from a stream, existing water supply, or from artesian wells is perhaps available.[1]

After the water has performed its duty as a condensing agent, it is considerably higher in temperature, and whether it is allowed to pass away as drainage or be cooled and returned to the reservoir will depend on the supply available and its cost. In any case, traces of oil will be found to contaminate it, and the water returning from each section of the plant must pass through a separator, designed to recover any oil, before passing into the main drain, which should also be provided with an efficient separator at a point just before the water enters the reservoir. Fig. 39 illustrates a large separator, and the same design may be reduced to suit each section.

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[1] Dr P. F. Frankland's article on "Water" in Thorpe's *Dictionary of Applied Chemistry*, 1913.

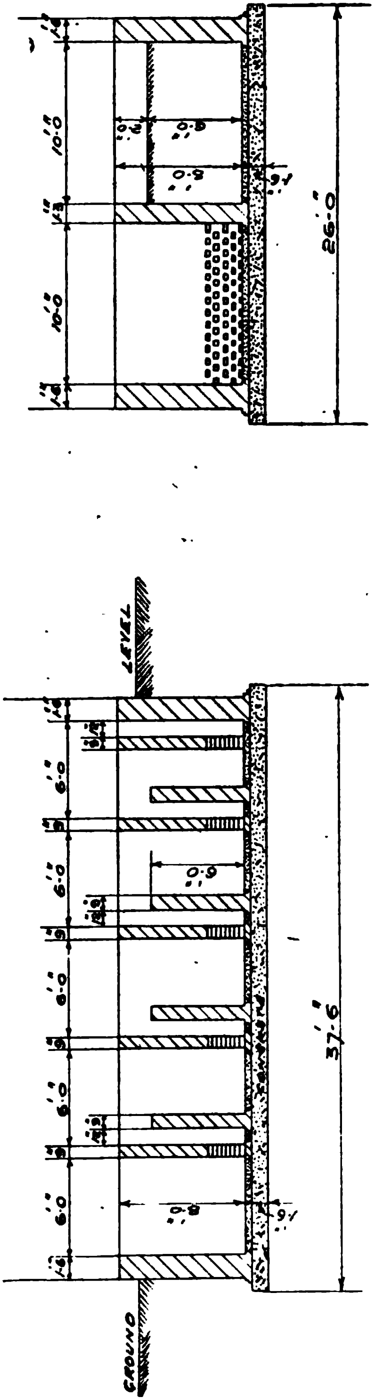


Fig. 39.—Works separator.

The quantity of condensing water for the whole refinery will be arrived at by calculating the amount required for the individual units.

Condensed steam separated from the distillates, at the tail-houses or worm ends, will return along with the condensing water.

Water employed for chemical treatments will be contaminated with salts removed from the oils, and after separation of any oil carried off will not return to the reservoir but pass away as drainage.

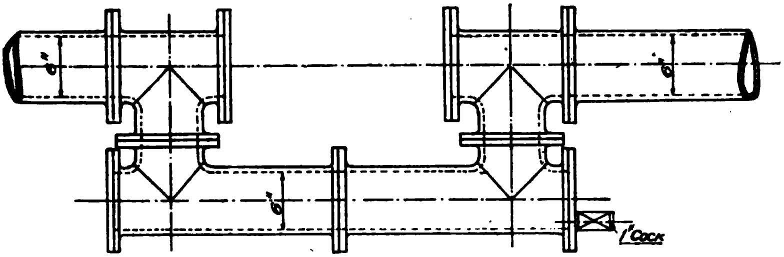
The disposal of tar precipitated from the oil by the sulphuric acid treatment requires consideration. The practice in Scotch refineries is to wash the free acid from the tar by steaming and use it as fuel. The acid washings will be neutralised with the excess soda water from the chemical treatments before discharge into the effluent.

Consideration of these points will show that it is necessary to consider, as already said, the questions of condensing water and drainage conjointly.

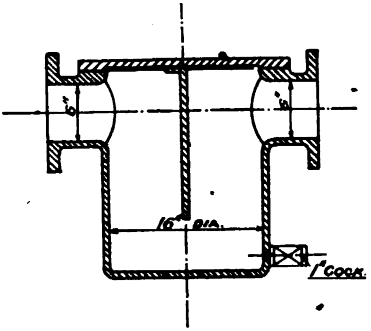
Drains are built of brick, or concrete, faced with cement and laid at proper level throughout. The overflow from the different sections should pass through a fire-trap (fig. 40) before joining the main drain. The object of this is to prevent, in event of fire, the burning oil flowing into the main drain. Fire-traps interposed at various points throughout the whole drainage system will prevent this.

If the return condensing water must be cooled, the drains are built wide and shallow to allow of evaporation, and it may even be imperative to introduce a cooling tower into the system.

Fire Precautions and Appliances.—The best precaution to take against fire is to construct the refinery on sound engineering principles, allowing nothing flimsy in the construction. The departments dealing with low-flashing fractions must be isolated, and that in which petrol is dealt with conducted as if it was an explosive factory.



Fire-trap made up of pipes, with blank flanges for cleaning.



Fire-trap, with cover for cleaning.

Fig. 40.

Smoking or naked lights are not permissible in any part of the refinery except in the rooms specially reserved for the purpose. It is best to arrange buildings at reasonable distances apart, to prevent, as far as possible, fire occurring in one department spreading to another.

Several tons of sand kept convenient to all stills and here and there over still benches in small quantities in buckets is a wise arrangement. Fires occurring in stills are mostly those caused through the breaking of a joint, and a few handfuls of sand thrown at the moment of outbreak may prevent it growing to larger dimensions.

Water hydrants must be connected, at numerous and convenient

points throughout the whole refinery, to the water service, and hose and nozzles kept in boxes close by. These must be constantly tested and maintained at full efficiency. Water is of no use in extinguishing burning oil in large quantities, but many a fire taking place at a still manhole or other joint has been wiped out by a well-directed stream from a fire nozzle at 50 to 60 lbs. pressure per square inch. In tank fires, too, water is useful in keeping down the temperature of neighbouring tanks. The whole refinery staff should be trained in fire drill, and a fire brigade established whose sole duty is the maintenance of the appliances, consisting of hose, nozzles, hydrants for water, shovels, and buckets or baskets for sand—in fact, keeping all the appliances ready for use at a moment's notice. In large establishments it is a common practice to erect a "fire" tower at a sufficient elevation to admit of a clear view of the whole premises, and from which a vigilant watch may be kept.

When tanks of small capacity become ignited, the contents may be rapidly pumped away and the duration of the fire thus shortened. Whether the contents of tanks of large capacity may be salved or not will depend on the rate at which they can be pumped away. Within the last few years suggestions have been made for extinguishing tank fires by means of carbonic acid gas (CO_2) generated by mixing sodium bicarbonate with sulphuric acid, and also by pumping frothy mixtures, impregnated with carbonic acid, on the surface of the burning liquid. Whether these suggestions have proved successful or not, in actual practice, on the large scale, the writer has not found out, although there are records of one or more being quite successfully employed experimentally.

Hand grenades, while of little value when a fire has actually taken hold, have frequently, when used just at the moment of outbreak, prevented a more serious conflagration.

Steam is useful in extinguishing fires in small tanks and buildings.

Prevention, however, is better than cure, and in a well-arranged refinery the risk will be reduced to a minimum. An important point to bear in mind is to retain as little light oil as possible in receiving tanks at stills, pumping it away to the sections where it is to be specially dealt with at regular periods. Figs. 41 and 42 show a 1,000,000 gallon tank on fire and the débris left after the oil was consumed.

Housing of Employees.—This will depend entirely on the situation of the refinery. Accommodation may be required for all, from the works manager down, and the question can only be considered when the conditions existing at the site are known.

Effect of Petroleum on Health.—In the writer's experience, workmen have not suffered from ill-effects through working with petroleum and its products. Workers in the shale oil industry suffer from an eruption known as "paraffin boils," but this is confined to those employed in the paraffin plant, and a change of employment usually affords relief. In the industries in which the writer has gained many years' experience, no such ill-effects were observed.

Intoxication and even suffocation have resulted through men entering tanks containing light vapours, but this may be prevented by thoroughly ventilating the tank before the workman enters it, or by providing him with a suitable form of "gas helmet." Mr Herbert Barringer, M.Inst.E.E.,[1] recorded a case where a repair was effected by a diver who descended into a tank containing liquid petroleum in a diving bell.

Layout of Plant.—In arranging the plant on a selected site it must be decided whether or not operations are likely to be extended at a later period on that site. If extension is probable, this must be borne in mind when laying out the initial installation. The relation of the site to transport facilities, whether by railway or water, must be considered carefully. For instance, if coal is to be used as fuel, and carried to the refinery by railway, the furnaces consuming it will be arranged at points necessitating least haulage. The best site, in the writer's opinion, is an undulating or sloping one on which the plant may be arranged to allow the oil to flow by gravity from one department to another with little or no expenditure for pumping plant. The worst is one which is perfectly flat, the character of the ground preventing receiving tanks being sunk to a low level, thus necessitating pumping at every stage of the process. An undulating or sloping site assists drainage, which, on a level site, in certain positions, may be troublesome. It is not always possible to select an ideal site at the locality where it is desirous to erect a refinery, and the arrangement of the plant must therefore be made to suit existing conditions.

Each section of the plant must be divided from its neighbour by a reasonable space, however, to ensure as far as possible non-interference with the operation of the one should trouble occur in the other.

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[1] "Oil Storage," a paper read before the Institution of Petroleum Technologists, London, on 18th January 1916.

CHAPTER III.

STORAGE TANKS.

The number of tanks required for crude oil storage, and for finished products ready for delivery to the market, and their individual capacity, will depend on circumstances. If, for instance, the refinery be situated in reasonably close proximity to the oil-fields, then crude oil tankage may not be required for the refinery *per se* at all, or only on a very limited scale. If, however, the refinery be separated from the oil-fields by considerable distances, either of water or land, then, obviously, provision must be made for possible longer or shorter periods in which the connecting water transport or pipe-lines may be out of commission. Generally, as a minimum, in circumstances other than close proximity of the refinery to the oil-fields, it will be well to provide at least a couple of months' crude oil supply. Once the daily throughput is decided, it will be a simple matter to calculate the total capacity required. Take 100,000 imperial gallons daily as the proposed throughput ; making the above provision, then, in round figures, 6,000,000 gallons storage room will be necessary. Three 2,000,000 gallon tanks will accommodate that

TABLE VI.—TANKS.

Products.	Per cent.	Monthly yield.	Number of tanks.	Individual capacity.
		gallons	gallons	
Petrol	6	180,000	2	200,000
Benzine	16	480,000	2	500,000
Kerosene	35	1,050,000	2	1,000,000
Intermediate oil	10	300,000	2	300,000
Light lubricants	10	300,000	..	..
Grade I.	..	..	4	50,000
„ II.	..	..	4	
„ III.	..	..	4	
Heavy lubricants	10	300,000	..	..
Grade I.	..	..	4	50,000
„ II.	..	..	4	
„ III.	..	..	4	

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quantity, and, besides being cheaper than the equivalent volume in smaller tanks, will occupy less area for erection. Also allow two months' capacity for storage of liquid products, on the basis of the above yields, and the capacity and tankage shown will provide comfortable working space if stocks are maintained equal to about four weeks' production.

Chapter IX., "Engineering Specifications," has been compiled by Mr John Gillespie, M.I.M.E., a petroleum engineer of long and wide experience, and in it will be found specifications of all the above-mentioned tanks, as well as for capacities of 100,000, 20,000, 10,000, 5000, and 2000 gallons, those for the last three being both upright and horizontal.

British refiners have not adopted concrete reservoirs for the storage of petroleum.

The construction of steel tanks is undertaken by many British firms. Foundations must be designed to suit the particular conditions of the site selected, but all tanks of large area are laid flat on their foundations without brickwork. A common practice is to build on a foundation of rammed sand, surrounded by a retaining wall. For volatile oils—petrol or crude oil containing petrol—the tanks should be fitted with gas-tight tops, slight pressure being maintained by means of a regulating arrangement in the form of a gasometer. Whether the whole group or individual tanks are to be enclosed by bunds, as a safeguard to surrounding property, must be considered. The danger does not lie so much in the possible collapse or rupture of the tank itself in case of conflagration or other misfortune, as in the breaking of connections letting loose the contents. It is a good plan when arranging pumping connections to make provision to enable the contents of the tank, to which accident has occurred, to be pumped into another.

If the site necessitates bunding, the material of which the bunds are to be constructed will require to be considered. If of earthwork, there must be regular inspection to prevent leakage except through the proper outlets for drainage. Whatever material the bunds are constructed of, the inner area must be cleared of drainage through efficient separators, no oil being allowed to escape. If bunds are dispensed with, care must be taken to place the tanks in such a position that any great, or small, leakage due to accident will flow to a lower level, where it will be retained without damaging surrounding property, and where it may burn itself out if on fire.

As with ironwork generally, so also with steel tanks, protection from the influence of the weather should be ensured by painting immediately after erection and repainting at regular necessary intervals. Petroleum-resisting composition (shellac varnish) is frequently used for protecting the inside of the top plates should the vapours have a corrosive action. Volatile oils are best filled through the connection at the bottom of the tank, thus creating as little disturbance of the oil, during pumping, as possible. Swing suction pipes for discharging are frequently fitted to prevent any dirt or water being drawn off—a very necessary precaution both as regards crude oil entering the refining plant or finished oils under despatch.

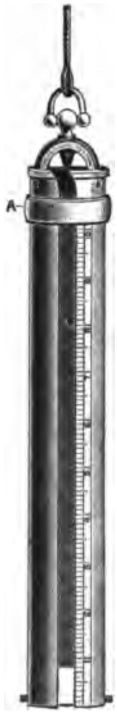


FIG. 43.—Redwood-Barringer water-finder.

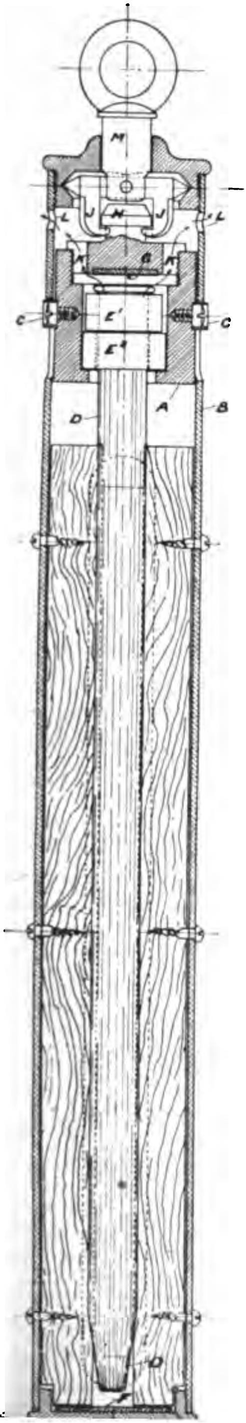
Precaution must also be taken against atmospheric electric discharge, and it seems generally agreed that the best means to this end is to attach the conductor points to the hand-rail usually fitted to tank roofs. The tank is earthed through its contact with the ground on which it is built, no wiring being necessary. Pumping of volatile oils in bulk should be arrested during electrical disturbance of the atmosphere.

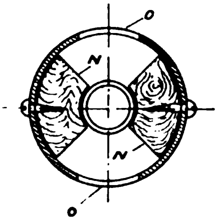
Provision made for cleaning tanks when sediment is present in the crude oil will save trouble after the plant has been some time in operation.

Tanks are built in plates riveted together either by hand or power. Oxy-acetylene gas and electric welding will, no doubt, play important parts in tank construction in the future.

For the measurement of contents, the sounding apparatus introduced by Sir Boverton Redwood and his brother, Mr Robert Redwood, is usually employed. Regarding it, the former in his *Treatise* says: "This consists of a suitably loaded steel tape, on which, at about the level of the oil, an ebony plate is clipped. As the oil level is very clearly defined on the surface of the ebony indicator, accurate measurements can thus be taken; and the appliances possess other obvious advantages over the rigid sounding-rod previously employed."

In refineries, should water be present with the crude oil, it is simply run off at the drain-cock on the bottom of the tank after it settles out. The same is done if water should, through carelessness or accident, be found in tanks containing products. At the time of making delivery of large quantities it may be necessary to show the exact amount, and in this case the Redwood-Barringer registering water-finder will be found a useful instrument. It is described as follows:—"It depends for its action upon a change effected by water on the surface of a chemically-prepared paper upon which oil has no action, and consists of a weighted hollow brass cylinder provided with a convenient means of suspension in a vertical


M
N
J
J
L
L
K
G
K
C
E'
C
E²
A
B
D
D
F


O
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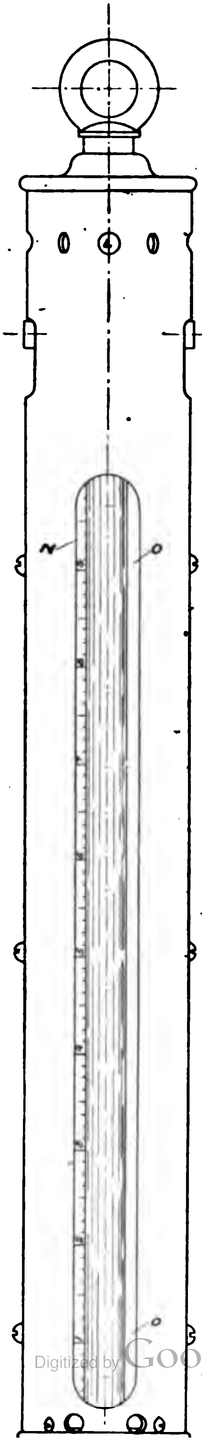
N
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O

position. This collar holds in position a strip of the prepared paper, which lies in two longitudinal grooves cut on opposite sides of the cylinder. At each side of each groove, a scale, divided into inches and tenths, is engraved on the cylinder. The apparatus is lowered into the tank by a cord, and when it touches the bottom, is allowed to remain in a vertical position for a sufficient time for the water to act upon the prepared surface—usually about thirty seconds. On withdrawing the instrument, the depth of water is found indicated on the paper on both sides. The illustration exhibits the appearance of the apparatus after use, the paper registering a depth of eight-tenths of an inch of water" (fig. 43).

The Simmons' water-finder (fig. 44) registers the depth of water without the use of chemically-prepared paper, as will be followed from reference to the descriptive illustrations.

To operate the instrument the weight, A, is raised inside the casing, B, by the said projections or trunnions, C, the glass tube, D, is then suspended by the soft rings, E¹ and E², E¹ being cemented to top of tube and rests upon E² fixed to annular opening in weight, A, leaving a space between the lower end of glass tube, D, and the bottom pad, F, to admit the oil or water which pass out through the spaces above the glass tube, D, and the top pad, G, escaping through the holes, K, in the top of the weight, A, and through the holes, L, in the casing, B.

The instrument, when suspended by the eye-bolt, M, is lowered into the tank, the oil and water pass through the tube, and on reaching the bottom, the clutches, J, release themselves from the stud, H, allowing the weight, A, to fall and close the tube top and bottom, and on pulling on the eye-bolt, M, to raise the instrument, the clutches, J, pass over the stud, H, pressing the weight, A, downwards, and making a secure joint on the ends of the glass tube, D, until the instrument is raised and the amount of water read off on the scale, N, through the long slots, O, in the casing, B.

The capacity of each tank is carefully calculated and registered.[1]

The most recent important article on petroleum storage is that by Mr Herbert Barringer, already referred to.

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[1] Very useful tables for the calculation of deliveries will be found in *Petroleum Tables*, by William Davies (Dunn, Collins & Co.), 5th Ed.

CHAPTER IV.

DISTILLATION.

THE enormous increase in the demand for fuel oil during recent years has influenced the design of distillation plant for crude oil. Residues, hitherto of little or no market value and therefore consumed as fuel in the refinery, or associated works, in a more or less extravagant manner, have become valuable products.

The main objective, at the present time, is to perform crude oil distillation with the lowest possible fuel consumption, in plant of the utmost capacity, while reducing, if not entirely eliminating, redistillations of once-run fractions. Considerable success has attended efforts in this direction, as will be seen from perusal of the following pages.

Crude Oil Distillation.—For the removal of low-boiling fractions only, the form of continuous still known as a tower still is cheap in constructional and working cost. It is heated by steam, generated under 30–40 lbs. pressure per square inch and maintained in a dry condition by passage through a heater before entering the still. To remove 10 per cent. to 12 per cent. from a crude oil containing 20 per cent. to 25 per cent. boiling under 150° C., about 1 per cent. of liquid fuel is required, the consumption being calculated on the volume of crude oil entering the still. This may be reduced by the addition of preheaters, the functions of which are afterwards explained. The plant illustrated by the diagram (fig. 45) is not equipped with these appliances, but several of various designs are suitable for connecting with it, if so desired. The still is an insulated vertical cylinder, fitted with perforated plates resting on coils through which the steam is conveyed to the bottom, where it enters. These coils serve the double purpose of carrying the steam to its inlet and heating the oil in process of distillation. The steam on entering the cylinder ascends, meeting the crude oil as it descends from plate to plate, in a regulated stream, and carries with it to the condenser the light fraction, the removal of which is the object of the operation. The steam pressure indicated will not unduly increase the amount of mechanical "carry over," any contamination of the distillate arising from the commotion in the still being rectified by passing the vapours through an atmospheric condenser, the condensate thrown down returning to the still.

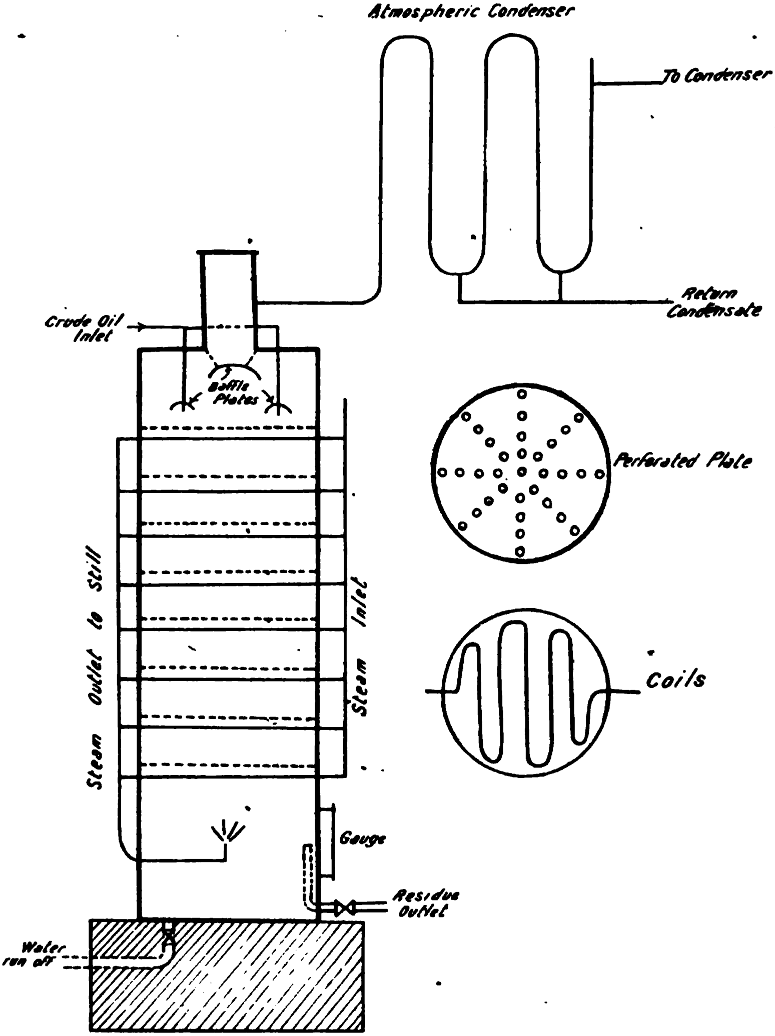

FIG. 45.—Topping still.

A combination of this plant with fractionating preheaters is illustrated by fig. 46. In this arrangement steam heating is superseded by direct firing. The crude oil enters at y_1, the residue preheater, D, which is heated by the residue flowing from the still, B. Overflowing from D, it passes through the outer shell of the dephleg-

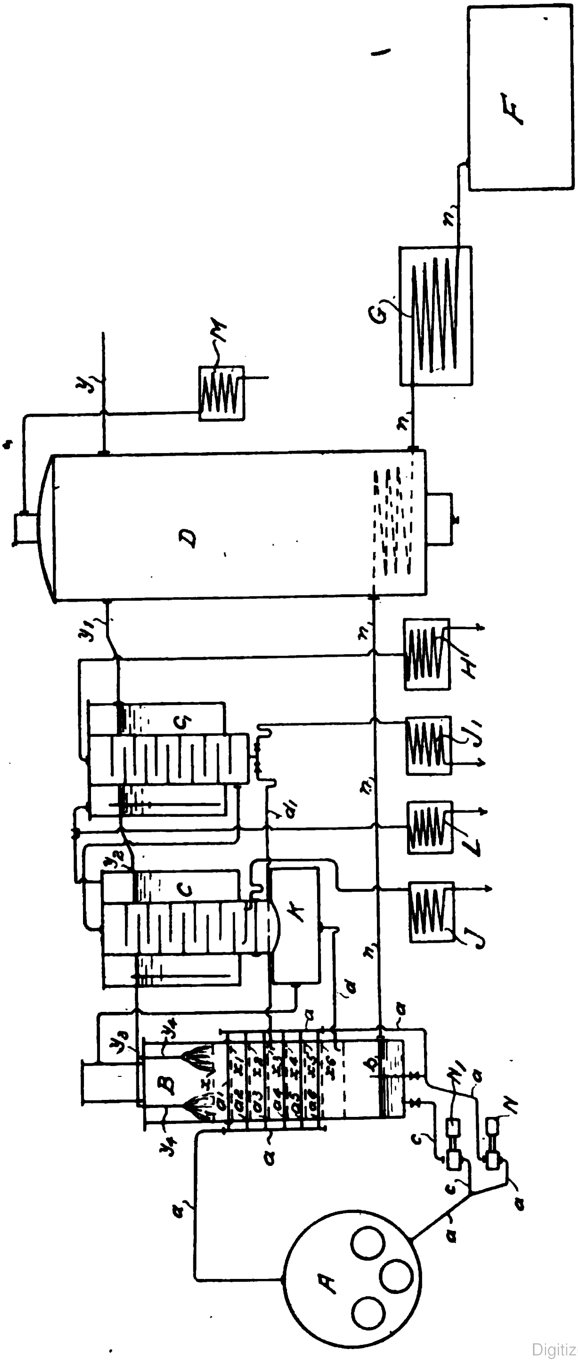

FIG. 46.—Topping still with fractionating preheaters.

mators, C and C_1, to B, which is identical with fig. 45. A portion of the residue collecting at the bottom of the tower is drawn off by the pump, N, into the vessel, A, where it is heated to the desired temperature under pressure and thence through the heating coils, x_1 to x_6, on which the perforated plates, a_1 to a_6, rest, finally returning to the heating vessel. y_1, y_2, y_3, and y_4 show the course of the crude oil through the system. M is a condenser for vapour distilling off preheater, D, and L a joint condenser for that coming off the crude oil surrounding C and C_1. The still vapours uncondensed in the latter pass through the water-cooled condenser, H, while J and J_1 are coolers for the condensates therefrom. K is a trap from which any "carry over" is returned to the still, and G a water cooler in which the residue may be further reduced in temperature before passing to the receiver, F. Steam is introduced into B to aid the separation of the distillate if necessary.

A departure in distillation plant is that patented a few years ago by Mr M. J. Trumble, a Californian chemist. Fig. 47 shows that the crude oil is heated to the desired temperature in pipes, or retorts, set in a primary furnace, the hot gases of combustion from which are utilised to heat the distillation chamber proper. This is a vertical cylinder, and the oil entering it at the top spreads over and through perforated plates, falling over a cone-shaped imperforate plate which diverts the continuous stream to the heated sides of the cylinder down which it flows in a thin film. Other conical plates are arranged at intervals beneath to maintain the flow in the desired channel until it reaches the bottom where the outlet is situated. Steam may be employed to assist evaporation, and the vapours are led into a vertical column and thence to condensing apparatus. The development of this system has been very rapid, as will be seen from the following abstract from Mr Arthur F. L. Bell's paper, "The Important Topping Plants of California."[1] Under the heading "The Shell Company of California," Mr Bell states that the economical capacity of the plant is 5000 barrels of crude oil daily, that many improvements have been made, and then proceeds as follows:—

"Besides the main topping plant, the Shell Company has erected a smaller Trumble retort with its evaporating tower for the purpose of running asphaltum and doing other experimental work. This part of the plant is not complete, and is not otherwise referred to in this paper.

"Like the Vernon plant, all condensing and cooling will be done in tubular condensers.

"The process will be carried on as follows:—The cold crude enters first the two vapour condensers, E and U (fig. 48), then condenser, E, where the distillates are condensed and returned to the lower compartment (N) of the separator, B (fig. 49), the process of evaporation being the same as described in the General Petroleum Company's Trumble plant. (The distillate from condenser, E, flows to the separator, B, entering the bottom compartment at one corner, travelling forward and backward six times through the series of compartments, N, heated by the hot vapours coming from the evaporator, A. As the distillates flow through these heated compartments, N, they are evaporated, the vapours rising through openings into either of the two upper compartments, P and R, from each of which the vapours are led to two independent condensers. The outside upper portions of the separators, B and C, are water jacketed, and as the hot vapours rise in the compartments, P and R, any heavy distillate, carried by the vapours, is condensed and drops back into compartment, N, so that only the lighter vapours escape from the respective compartments. The unevaporated distillates pass on to separator, C, where they are treated in the same manner as in separator, B, the vapours being conducted to two independent condensers.)

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[1] *Journal of the Institution of American Mining Engineers*, September 1915.

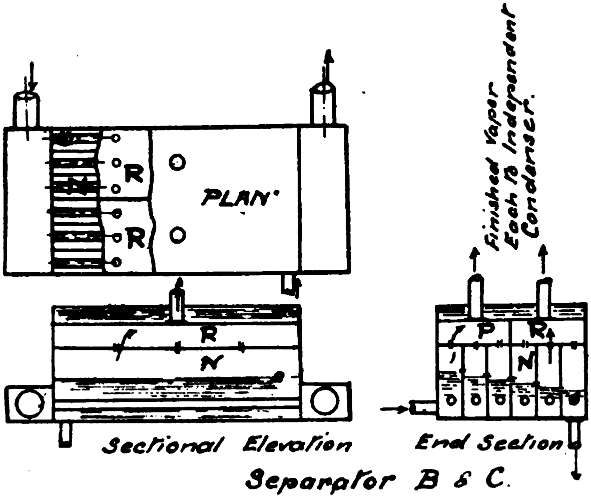

Fig. 49.—Trumble still separator.

"The vapours on leaving separator, B, which will have the lightest gravity, are condensed in condenser, F, and then pass off through jacketed pipe coolers to storage, while the unevaporated distillates from separator, B, pass on to separator, C.

"The second vapours coming off from evaporator, A, are collected in the central perforated pipe and led to the jacket of the evaporating column, T; after imparting a certain amount of heat to the inner shell, they pass through the heating tubes in separator, C, then to condenser, U, the condensed distillates being returned to compartment, N, of the separator, C, to be evaporated as previously described. The vapours then flow to condenser, G. These distillates are of a heavier grade than those from separator, B. The unevaporated distillates flow from separator, C, to separator, S, which is heated by the hot residuum passing through it on its way to the exchanger.

"The vapours from separator, S, pass off to the condenser, V, in the same manner as those from B to C; they are heavier than those from the other two separators. The unevaporated distillate from separator, S, is not further refined and is run to storage.

"Each of the three separators has two separate vapour chambers, each having a separate outlet connected to its independent condenser, so that the three separators will give six different cuts of varying gravity of re-run products. An additional product will be the still bottoms drawn off separator, S. The Shell Company is designing an additional Trumble plant of equal capacity, to be erected alongside the present one, in which still further improvements are contemplated."

It is therefore evident that this system of distillation will remove a large percentage from the crude oil, and in one operation; the arrangement of evaporators and exchangers described separating that distillate into several distinct fractions without recourse to redistillation. The initial cost of the plant, cost of operation and fuel consumption for a given throughput of crude oil and distillate, is infinitely less than that incurred by the installation of the long-established system, hereafter described, for distilling crude petroleum and for redistilling once-run distillates. The development of the Trumble system, or any modification of it, to the heavier portions of crude petroleum will be of special interest to refiners, for the objects in view are now of vital importance to the industry.

Another arrangement for the fractionation of petroleum distillates is the atmospheric condenser, developed by Mr Hugh L. Allan, which may be connected with either vertical topping or continuous boiler stills. It consists of a series of upright condenser pipes, each couple terminating in a vessel in which the condensate is trapped. The vapours pass from vessel to vessel, depositing in each a condensate becoming progressively lighter in specific gravity as the uncondensed vapours reach the water condenser. A spray of water may be employed to cool the limbs of the condenser if desired, and the vessels in which the condensate is collected are maintained by means of internal steam coils at regulated temperatures adjusted to suit the products to be obtained. Fig. 50 illustrates one section of the apparatus, a group forming the complete arrangement, for which patents have been applied for.

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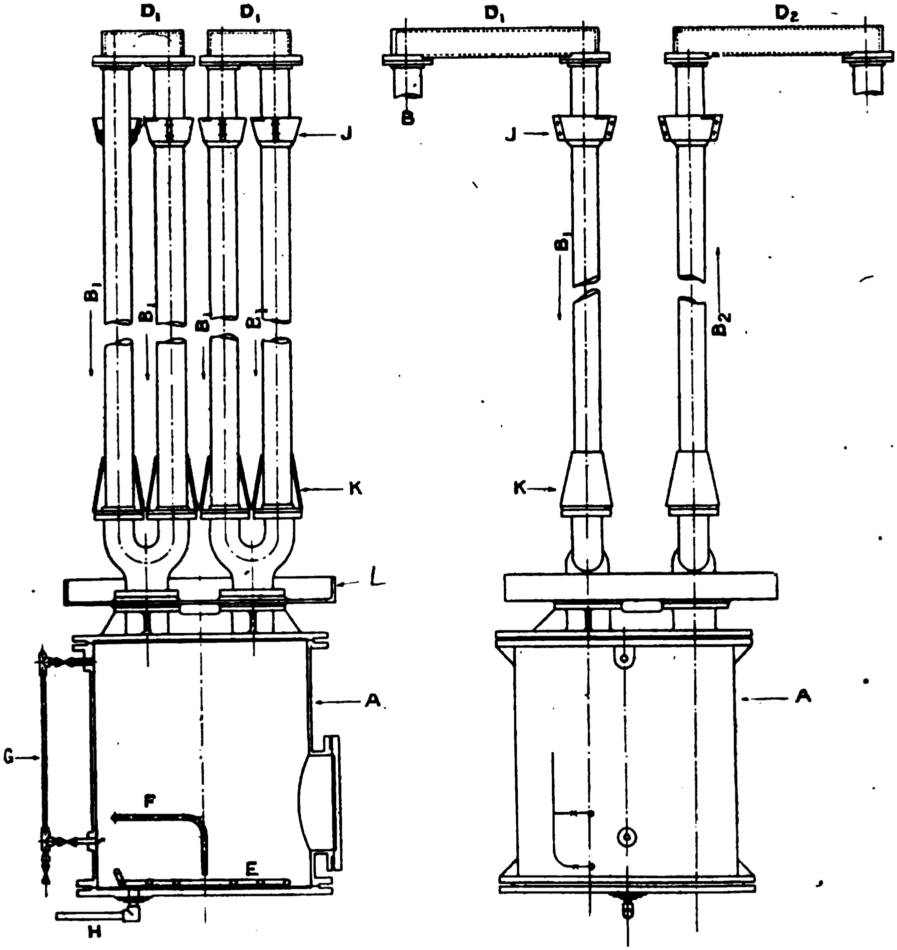
When 60 per cent. to 70 per cent., comprising all the benzine, kerosene, and perhaps intermediate fractions, are to be removed, it has been, and is common practice to distil the crude oil in a series of horizontal cylindrical stills, connected to distil continuously.



A = Cylindrical vessel.
B = Vapour inlet.
B_1 and B_2 = Malleable iron condenser pipes.
D_1 D_2 = Connecting box.
E = Closed heating coil.
F = Open steam pipe.
G = Gauge glass.
H = Run-off pipe.
J = Water distribution.
K = Anti-splash cone.
L = Water tray.

Gas from the still or stills enters at B. Passes through D *via* B_1 to A, and from A through B_2 to D_2 and on to the adjoining unit.

FIG. 50.—Allan's apparatus for continuous fractionation and dephlegmation of petroleum or other hydrocarbons, or mixture of volatile liquids.

Some refiners "top" the light fractions from the crude oil before it enters the cylindrical stills, but unless there exists some special reason for this complication it would appear that the whole distillation is best undertaken in cylindrical stills, without extra attention and loss of heat from the large volume of residue yielded by the topping plant.

The best known British system is that designed and patented by the late Mr Norman M. Henderson, Works Manager of the Broxburn Oil Co., Ltd., in 1883, for the distillation of shale oil, and since adopted by many refiners of petroleum. The stills were chiefly cylindrical in form, although those of some of the smaller units were wagon-shaped.

The crude oil flows from a charging tank erected at a suitable elevation, through a preheater, warmed by the passage of vapours from the second or third still, and thence into the first still. Here it is raised to distillation temperature, and the specific gravity of the distillate at the worm end of the condenser fixed. The feed of the crude oil is regulated, and the residue formed allowed to flow through a connection at the bottom into the second still at the top, and led from back to front to ensure the inlet and outlet being as far apart as the length of the still permits. In this still the oil is raised to a higher temperature, yielding a distillate of higher specific gravity, and so the oil passes through all the stills in series until the point is reached at which all the once-run benzine, kerosene, and intermediate oils are removed.

All the outlets for residues are provided with safety plugs to prevent the outflow of the still contents in event of accident to connecting pipes.

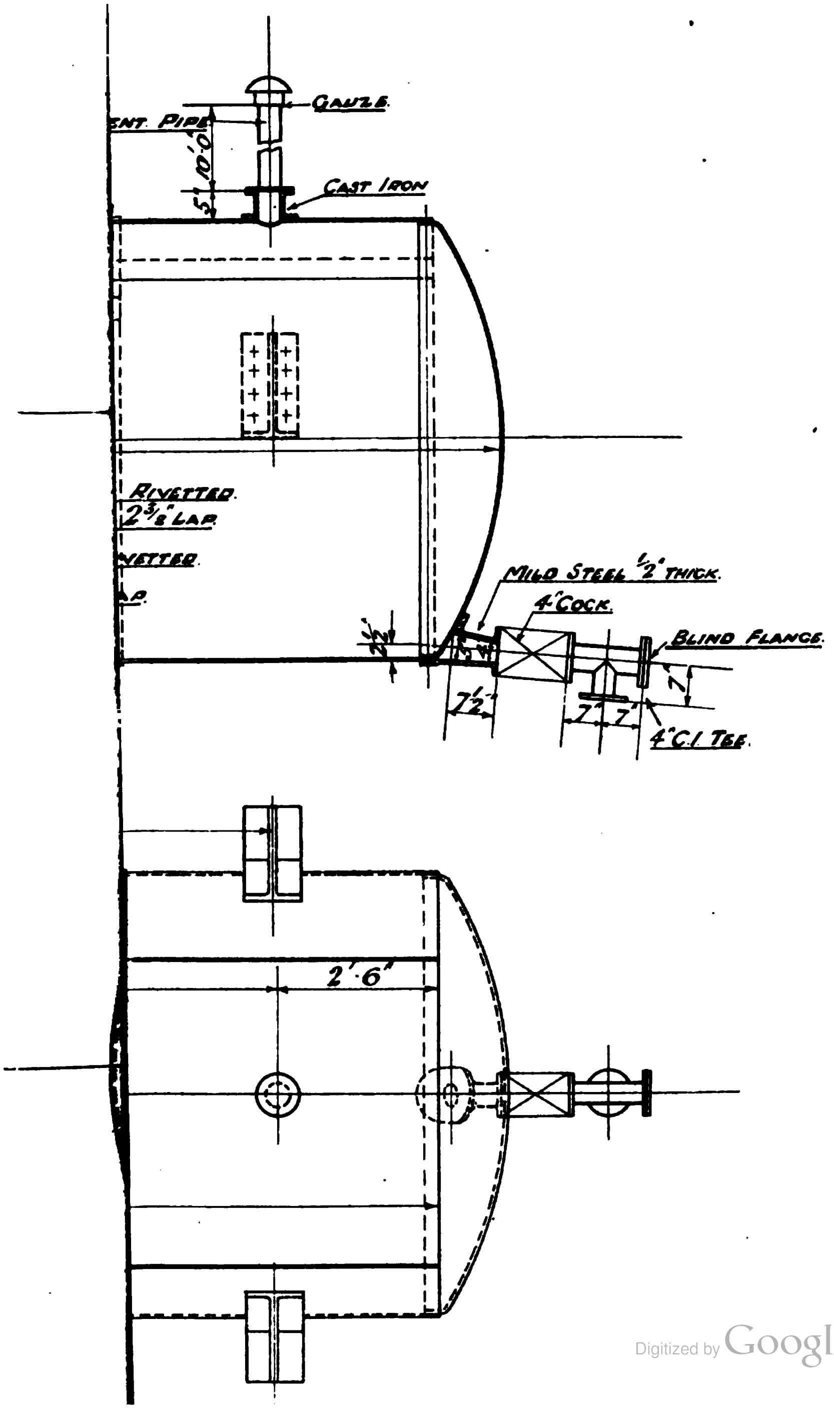
The residue, now containing only the heavier fractions, passes to one or other of a group of coking stills, worked intermittently in rotation and, in some instances, under partial vacuum. Here it is reduced to dryness, coke, or to a heavy residuum which may be burned as fuel.

The standard dimensions of Henderson's cylindrical stills are 19 feet in length by 7 feet in diameter. As many as eight have been worked in series, distilling approximately as much as 70 per cent. from crude oil. For good fractionation a rate of distillation approximating one gallon of distillate per square foot of evaporating surface per hour should be maintained.

A current of steam is introduced to facilitate the separation of distillates and to prevent "cracking," and this is generated in boilers conveniently situated. The proportion of steam is measured at the worm end, and should approximate 25 per cent. of the total distillate received.

Fig. 51 is a plan, elevation and section of a plant designed on the

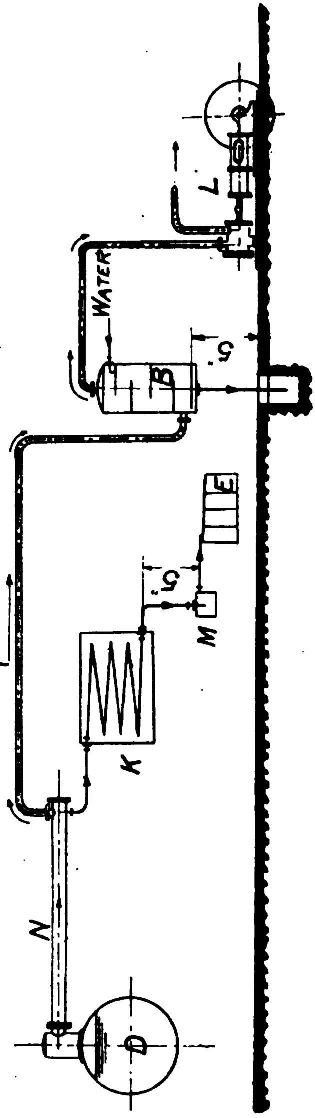
A.


GAUZE.
ENT. PIPE
5' 10'0"
CAST IRON
RIVETTED.
2 3/8" LAP.
VETTED.
AP.
MILD STEEL 1/2" THICK.
4" COCK.
BLIND FLANGE.
1/2"
3/4"
7 1/2"
7"
7"
7"
4" C.I. TEE.
2'6"

regular, otherwise, if delay occurs, the throughput of the whole system will be reduced. The time taken for the complete operation of the pitch still usually aggregates about forty-eight hours.

When the oil contains only a small percentage of pitch, it may be decided to use a certain amount of the residue as fuel, and in that case stills of the above type will be employed but with modified cooling tanks. If, however, it is decided to reduce the residue to coke, a still with a cast-iron bottom, which is readily removed, is most suitable. Care must be exercised in firing during coking and in cooling previous to the removal of the coke, as errors in these respects result in shortening the period during which the bottom may be used without repairs or renewal. Figs. 61 and 62 are typical of cast-iron bottoms of stills as illustrated by fig. 63. Many of these stills are in use at the present time, and have been employed for years in the Scotch shale oil industry.

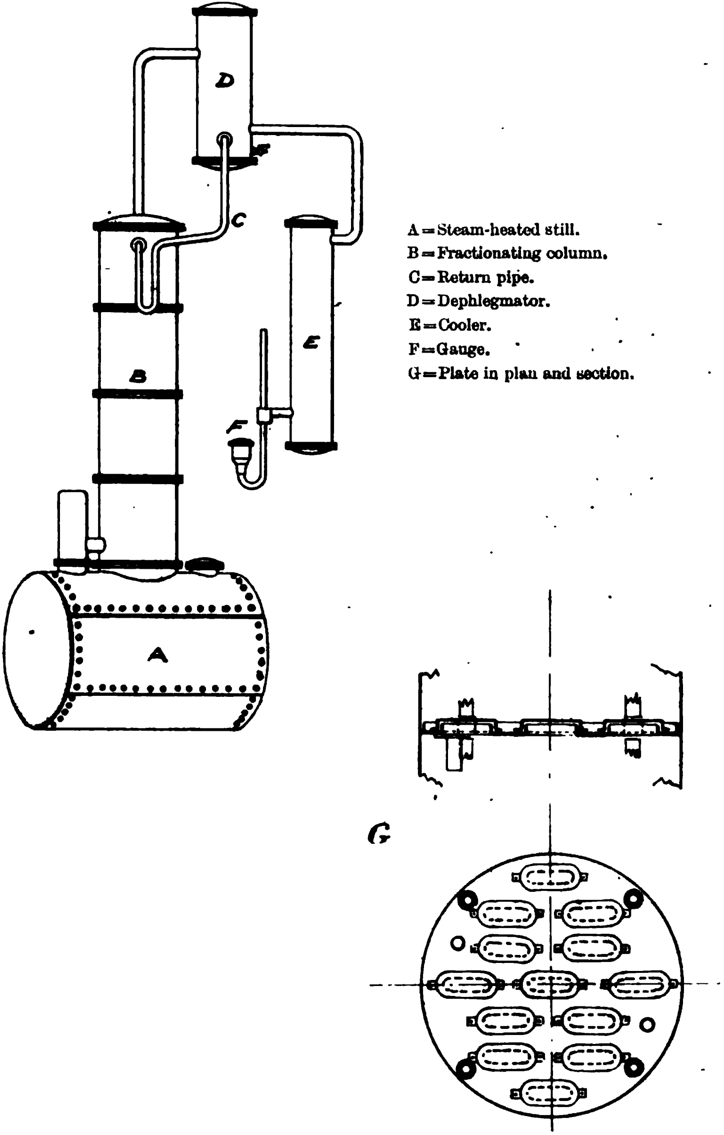
Heavy oil stills are frequently worked under partial vacuum maintained on the still by means of an air pump. The arrangement, which is applicable to any size or type of still, results in reducing the temperature of distillation 50° C. to 100° C. Figs. 63 and 64 illustrate the usual application of vacuum to heavy oil distillation.



D=Still; N=Air condenser; K=Cooler; M=Syphon; E=Tail-box; B=Barometric condenser; L=Air pumps

FIG. 64.—Scheme of distilling plant with low vacuum.

For the distillation, or concentration, of lubricating oil base, which is either a direct fraction distilled from the crude oil or the filtrate after the solid paraffin has been removed from such fraction, stills of exactly the same design as those described for manufacturing pitch or coke are used. In the distillation of high boiling-point fractions best results as regards colour and prevention of cracking are obtained when the operation is conducted in a current of superheated steam. This is controlled most satisfactorily by superheating in a separately fired furnace.



A = Steam-heated still.
B = Fractionating column.
C = Return pipe.
D = Dephlegmator.
E = Cooler.
F = Gauge.
G = Plate in plan and section.

FIG. 65.—Heckmann benzine still.

Redistillations of kerosene fractions are carried out in stills of the cylindrical type used for distilling crude oil.

The rectification of once-run benzine (redistillation to products of specified range of boiling-points) is conducted in steam-heated stills either of the continuous topping or cylindrical type. For close fractionation it is customary to employ a still fitted with a fractionating column either directly on the top of the still or adjacent to it.

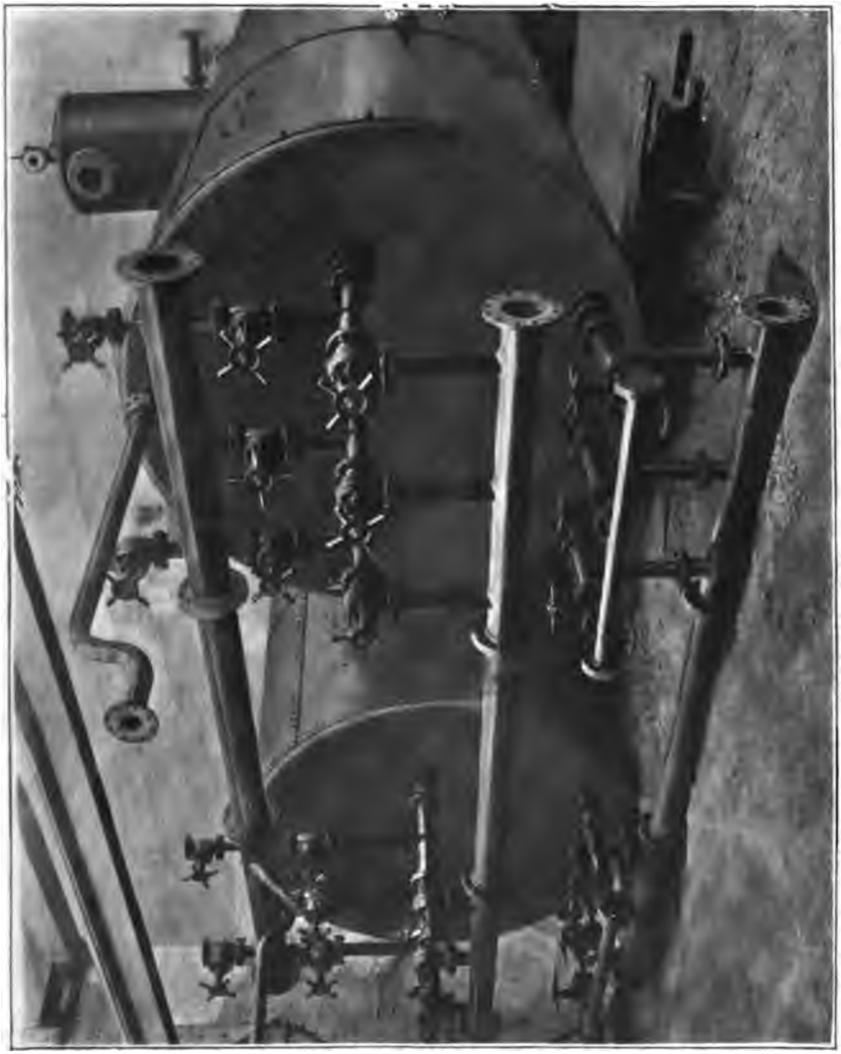


FIG. 66.—Two 10,000 gallon benzine stills, showing steam connections

There are numerous designs of fractionating columns, that illustrated by fig. 65—the Heckmann type—being well known as an efficient device. In other designs the arrangement of plates and the channels through which the vapours pass the descending condensate differ considerably. Some merely consist of a number of plates arranged to send the ascending vapour from the still through a continuous stream of condensate returning to it. This is the action in all columns, the object being to scrub the heavy and high boiling fractions from the ascending vapours, allowing only those of low boiling-point to pass over. Perhaps the cheapest form of fractionating column, and one giving excellent results, is that filled with pieces of pipe, nipples, tee-pieces, etc.

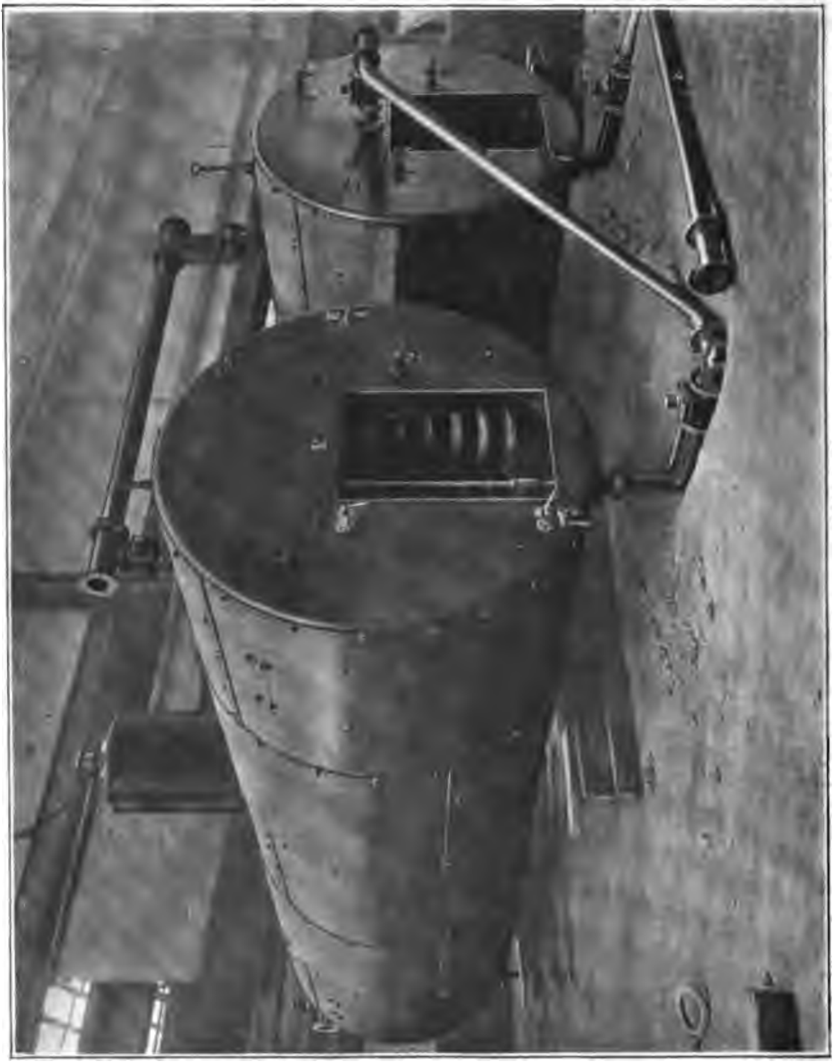


Fig. 67.—Two 10,000 gallon benzine stills, showing steam coils, benzine inlets and continuous connection.

For the ordinary petroleum proposition one redistillation of the once-run benzine will suffice to produce the desired "cut," provided a suitable column is used.

[illegible], p. [illegible]

The separation of the aromatic hydrocarbons from light petroleum distillates has attracted attention on account of the possibility of augmenting supplies of toluol for the manufacture of high explosives. For this the fractionation is more complicated, a number of redistillations being necessary to reach good concentration.

Regarding the dimensions of fractionating columns, or dephlegmators, Lunge[1] states: "The diameter of the fractionating column for benzol must, of course, vary with the size of the still. According to Spilker[2] it should be about 2 feet 8 inches for stills holding 5000 litres (=176·6 cubic feet), and 3 feet 3 inches for 10,000 litres (=353 cubic feet). Their efficiency increases with their size, especially if the number of the plates is correspondingly increased." These proportions seem excessive for petroleum work, however, because a 10,000-gallon still for rectifying once-run benzine, fitted with a 15-plate Heckmann column 5 feet 6 inches in diameter, gives excellent fractionation at the distillation rate of 500 to 600 gallons per hour, the whole charge being distilled, residue run down, and the still recharged ready for the succeeding run within twenty-four hours.

The still must be provided with internal coils of ample heating surface to ensure the proper rate of distillation, and hot residues, if available, may be used for preliminarily heating the contents of the still. Figs. 66 and 67 are illustrative of two benzine rectification stills in course of erection. The heating coils and connections are clearly shown.

The fuel consumed for redistillation of kerosene and benzine in the type of stills described is about 6 per cent. and 3 per cent. of liquid fuel by volume respectively.

Condensers, Coolers, and Preheaters.—The function of the condenser is to condense distillates issuing from the still in the condition of vapour into liquid. This is accomplished by circulating cold water through the tank containing a coil into which the vapours pass. The heat transferred from the hot vapours to the surrounding cold water is carried away by the latter. Coolers, working in connection with stills, are for the purpose of reducing liquids, either condensates or residues, to a lower temperature than that at which they are received from the stills. The cooling medium in this case is also water. A coil condenser, shown in detail on fig. 68, forms portion of fig. 63, and fig. 69 is a detailed drawing of the radiator condenser, which is a more compact design. The tubular form of coil condenser is also used, as is the atmospheric condenser. It will, however, be obvious that heat, dissipated into the atmosphere, is impossible of recovery.

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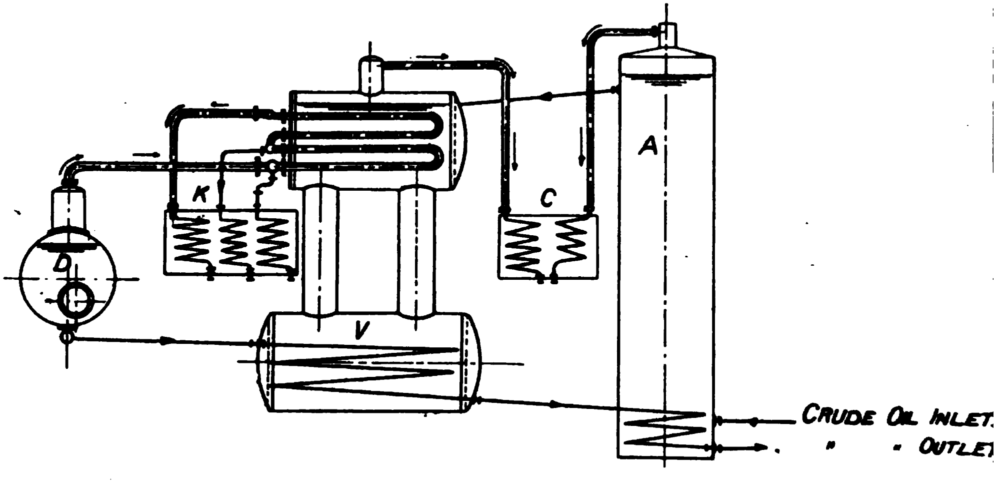
[1] Lunge's *Coal Tar and Ammonia*, 1916 Edition, Part II., p. 935.

[2] *Loc. cit.*, p. 60.

A distillate preheater performs partially the functions of the condenser, the cooling medium being oil instead of water. The heat imparted by the distillate, through the coil to the oil, raises the temperature of the latter, thus reducing the amount of fuel necessary to bring it to distillation point when it reaches the still. Since steam is employed during part of the distillation, the amount of heat recoverable by its condensation in the preheater is considerable. In the residue preheater a portion of the heat of the residue leaving the still is absorbed by the crude oil surrounding the coils through which the residue flows.



A = Vertical residue preheaters.
C = Benzine condenser for distillates of preheaters.
D = Still.
K = Cooler for 3 cuts from still.
V = Combined distillate and residue preheaters.

FIG. 70.—Scheme of intermittent distilling plant with crude oil preheaters.

Fig. 70 is illustrative of an intermittent still provided with preheaters. The design of the above condensing, cooling, or preheating apparatus is subject to the quantities and temperature of the distillates or residues entering them, and ample heat exchanging surface must be allowed. Rate of heat exchange is referred to later.

The subject of "preheating in petroleum operations" was discoursed at the Third International Petroleum Congress held at Bucharest in 1907, by Dr Kraemar, and the following is a free translation of his communication :—

"The question of preheating petroleum by means of its distillates or of the still residue, especially in regard to operations with Rumanian oil, has been the subject of a thorough investigation by Dr Weger in the laboratory of the Erkner Tar Distillery, and the results are here summarised.

"The material studied was a petroleum supplied by the Telega Oil Co., Ltd., Doftána, which, like the majority of the Roumanian oils, is relatively rich in benzine and kerosene and poor in paraffin.

"It was necessary to bear in mind that (1) in such oils the utilisation of the latent heat of evaporation of the whole distillate was restricted to certain limits to be considered in respect of the safety of the apparatus; and (2) the free naphthenic acids present in the Roumanian oil would act injuriously on metal, especially at the high boiling-points of the kerosene and solar-oil fractions.

"Consequently, Dr Weger limited his investigations to determining what advantages and disadvantages were connected with preheating, if, to begin with, the benzine fraction were carried over in great part, by naked or by coil-steam, continuously or intermittently, and the still-residue, thus completely deprived of water, were transferred to another vessel, and further exhausted by direct fire heat. Thereafter the study was extended to the utilisation of the latent heat of evaporation of the benzine, and of the heat of the benzine-less residue, and to the possibility of continuous distillation at determined temperatures obtainable by high-pressure steam. Continuous column-distillation was adopted, as is sometimes the practice in the tar industry, though generally continuous stills only are used for petroleum. Subsequent operations extended to facilitation of distillation by the use of naked steam, and finally the question was investigated whether or not a vacuum was desirable for the kerosene fraction.

"A. *Use of the latent heat of evaporation of the benzine.*—In the following formulæ—

B = the percentage by weight of benzine in the crude oil;
m = the initial temperature of the crude;
x = the required temperature in the preheated crude;
t = the temperature of the benzine vapour on entering the cooler;
Lb = the latent heat of evaporation of the benzine fraction under consideration;
Sb = the specific heat of the same; and, lastly,
Sr = the specific heat of the crude oil.

"Then, assuming complete interchange of heat without any loss, *i.e.* the benzine cooled to $m°$, we have the following equations:—

"Loss of heat by the benzine—

$$\mathrm{BLb} + \mathrm{BSb}\,(t - m).$$

"Gain of heat by the crude oil—

$$100\,\mathrm{Sr}\,(x - m).$$

"Combining these equal amounts of heat we find—

$$x=\frac{B}{100}\ \frac{Lb+Sb\ (t-m)}{Sr}+m.$$

"The values of Lb, Sb, and Sr are unknown, and for accurate work must be found by exact physical investigation. Approximately, Sb and Sr are equal, and may be taken at 0·5 as a mean, since—

Hexane,	boiling-point, 69° C.,	specific heat,	0·5042
Heptane,	,, 98° C.	,, ,,	0·4869
Octane,	,, 126° C.	,, ,,	0·5111
Decane,	,, 173°–174° C.	,, ,,	0·5058

Lb may be taken at 75 as mean, since—

Pentane,	boiling-point, 36·3° C.,	latent heat,	74·89
Hexane,	,, 69° C.	,, ,,	89·16
Decane,	,, 173°–174° C.	,, ,,	60·83

m will be about 15° C., and
t should be 120° C. from the subjoined data:

"(In the continuous column-distillation where vapours of the lower and higher boiling fractions pass over simultaneously, the mean temperature of the vapour is naturally lower than the final heat of still operation, *i.e.* $t=120°$, when the boiling-point of benzine is about 150°.)

"B, for the Telega crude, is about 25 per cent. by weight. Under these conditions $x=79°$ C.

"In practice, however, even with the best coolers, the heat-interchange is incomplete; the benzine is not cooled to $m°$, but to $m°+d°$, where *d* is a coefficient dependent on the construction of the cooler. Consequently, the crude oil is not so thoroughly preheated, and we have—

$$x=\frac{B}{100}\ \frac{Lb+Sb(t-[m+d])}{Sr}+m.$$

Taking *d* at 10°, we find $x=76°$.

"There is also a loss of heat outwards, lowering this figure again.

"Supposing, as investigation has established, that in heating the aforesaid Telega crude, the first drop of distillate falls into the cooler when the still temperature reaches 74°; that means, under the conditions mentioned, that distillation would just begin to be possible *in the cooler* by means of the latent heat of the benzine (being pure, with no kerosene), since here a temperature of 76° prevails. Attention must be given to this point in the construction of the cooler.

"All the above refers to distillation without naked steam. If this be used, its high latent heat will effect a still more important result. Taking the weight of distillate as ten times that of the steam, let Lw be the latent heat of steam (=536) and Sw that of water (=1).

Then—

$$\mathrm{BLb}+\frac{\mathrm{B}}{10}\mathrm{Lw}+\mathrm{BSb}(t-[m+d])+\frac{\mathrm{B}}{10}\mathrm{Sw}(t-[m+d])$$

is heat lost by vapour and steam, and 100 Sr $(x-m)$ is heat taken up by the crude oil. Thence $x=108°$.

"It must, however, be considered that in the utilisation of steam the temperature of the benzine vapour passing over may be substantially lower; in fact, to obtain a uniform benzine, t must be taken at about 90°. Then x will be 99°.

"By the lowering of t, therefore, the elevation due to the introduction of Lw and Sw is not exactly proportionate, since by using naked steam the crude in any case will be preheated to a higher temperature, certainly to a point above the initiation of boiling.

"B. *In the preheating of the crude oil by the discharged oil, after removal of the benzine*, a mixture of kerosene and residue, a heat interchange of the most uniform nature is effected.

"From 100 parts by weight of crude oil at 15°, we have 75 parts of topped oil at, say, 150°.

"The cooling of the topped oil is perhaps not generally thorough, and could be carried further; and 150° is not so high as to cause any difficulty whatever in transfer through pipes. But assuming that such transfer should not be effected at more than 70°, and that the specific heats of the topped and crude oils are identical (an assumption involving no great error), the crude can be heated from 15° to 75° with the topped oil.

"In practice, both systems are alternately used, preheating with the benzine vapour and with the topped oils. The Popelka-Veith patent combines them, and the cooler serves simultaneously as a still. There are greater drawbacks with the benzine than with the topped oils, the latter being the preferable system. Besides this, if the crude oil is to be at once distilled with high-pressure steam, both fire and steam are economised by preheating. For continuous column-distillation, preheating is unconditionally advantageous.

"To deal briefly with intermittent distillation the following experiment serves:—

"About 2 litres of crude were distilled in a still without column, and comparison made of the temperatures prevailing in the still and at the outlet. Tb and t respectively (boiler and exit).

Tb.	*t.*	Difference.
140°	118°	22°
150°	126°	24°
160°	133°	27°
170°	142°	28°
180°	150°	30°

"In order to remove all benzines up to boiling-point 150° by coil-steam, it was necessary to have the still temperature at least 180°, corresponding to a pressure of fully 10 atmospheres.

"In continuous distillation the conditions are so far more favourable that one has only to deal with steam at the lower mean temperature. This, of course, refers only to continuous column-distillation not to continuous still-work. The mean temperature must be about 120°, if the distillate is to have boiling-point 150°.

"On the other hand, some heat is, of course, lost in the long passage through the column, so that the still temperature, Tb, must be rather higher than 120° + 30°.

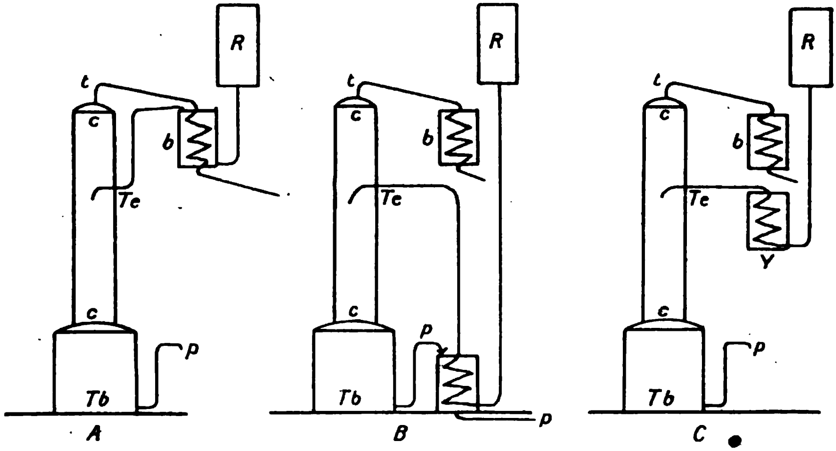


Fig. 71.—Preheating diagrams.

"In small apparatus the loss of heat was so serious that at Tb 180° the requisite 120° was never reached for *t*. (See experiment 2, below.) Of course, the initial temperature is a factor in this case.

"For continuous distillation the laboratory apparatus A and B were used at first (fig. 71).

"Although, from calculation, only a meagre preheating to 79° or 75° could be expected without using naked steam, it must yet be admitted that, despite the most perfect protection possible, in small apparatus the loss of heat outwards was too great, and the results indicated no adequate utilisation, the preheating proving yet much feebler.

"Then apparatus C (fig. 71) was used, in which the crude oil was brought to the desired initial temperature in a specially-heated preheater.

R = the crude oil reservoir ;
cc = column ;

b = benzine cooler ;
p = outlet of topped oil ;
Tb = still temperature ;
Te = temperature of preheated crude at entry to column ;
t = temperature of benzine vapour passing over.

"The following four experiments were carried out with apparatus C:—

"1. Without naked steam. Tb at about 150°, corresponding to a pressure of about 5 atmospheres.

Tb	156°	156°	152°	153°	154·5°	157°	158·5°	158°	mean 155°
Te	113°	113°	114°	114°	115°	108°	113·5°	113°	„ 113°
t	93°	81°	85°	87°	86°	90°	92°	88·5°	„ 88°

"The columns represent readings at thirty minutes apart.

"About 20 per cent. by volume was distilled off, giving a benzine of 0·7035 specific gravity with the following boiling-point :—

Up to 47°–50°	2·0 per cent.
„ 60°	8·0 „
„ 70°	23·0 „
„ 80°	42·5 „
„ 90°	63·0 „
„ 100°	80·0 „
„ 110°	82·5 „
„ 111°	90·0 „

"During the experiment there flowed off about 18 per cent. by volume of the mixture of oil and residue of specific gravity 0·877 and the following boiling-point :—

Up to 128°–130°	1·0 per cent.
„ 140°	4·5 „
„ 150°	9·5 „
„ 160°	14·0 „
„ 170°	18·5 „
„ 180°	22·5 „ etc.

"The mixture of oil and residue left in the still, approximately 60 per cent. by volume, had specific gravity 0·863 and the following boiling-point :—

Up to 123°–130°	3·0 per cent.
„ 140°	7·0 „
„ 150°	12·0 „
„ 160°	17·5 „
„ 170°	22·0 „
„ 180°	26·5 „ etc.

"That is, by not using naked steam, temperatures 155° Tb and 88° *t* were insufficient to eliminate all the benzine, the distillate boiling only up to 111°, whilst, on the other hand, the residual mixture stood at much too low, 128°—123°.

"2. Also without naked steam. Tb at 180°, corresponding to 10 atmospheres' pressure :—

Tb	175°	174°	175·5°	177°	178°	180°	176°	178°	178 5°	180°	177°
Te	114°	118°	115·5°	119°	119°	119°	112°	108°	112°	111°	119°
t	105°	100°	106·5°	109°	109°	110°	111°	103°	105°	107°	100°

Tb	176°	177°	180°	180°	178°	179°	176°	175°	176°	176°	177°	mean	177°
Te	114°	115°	115°	115°	116°	121°	118°	119°	118°	116°	112°	„	115°
t	100°	110°	112°	110°	102°	101°	106°	105°	108°	109°	108°	„	106°

"The columns represent readings at ten minutes apart.

"About 28 per cent. by volume passed over, giving a benzine of 0·722 specific gravity and the following boiling-point :—

Up to 57°—60°	1·5 per cent.
,, 70°	6·5 ,,
,, 80°	18·5 ,,
,, 90°	33·0 ,,
,, 100°	51·5 ,,
,, 110°	68·5 ,,
,, 120°	84·5 ,,
,, 130°	89·5 ,,
,, 131°	90·0 ,,

"During the experiment there flowed off about 30 per cent. by volume of the mixture of oil and residue of 0·895 specific gravity and the following boiling-point :—

Up to 150°—160°	4·5 per cent.
,, 170°	8·5 ,,
,, 180°	13·5 ,,
,, 190°	18·5 ,,
,, 200°	22·0 ,, etc.

"The mixture of oil and residue left in the still, about 42 per cent. by volume, had specific gravity 0·893 and the following boiling-point :—

Up to 146°—150°	2·0 per cent.
,, 160°	5·5 ,,
,, 170°	9·5 ,,
,, 180°	13·5 ,,
,, 190°	17·5 ,,
,, 200°	21·5 ,, etc.

"That is, again, with Tb at 177° and Te at 115°, the benzine was still rather too low-boiling.

"From these two trials it may be concluded that *t* must be about 120° if a sufficiently high-boiling benzine is desired, and the remainder sufficiently free from benzine. That is, Tb and Te must be somewhat higher.

"More advantageous work is effected by the help of naked steam.

"3. With naked steam. For 100 volumes of distillate, 18 volumes of condensed water from the steam. Tb at about 180°, corresponding to about 10 atmospheres' pressure :—

Tb . .	180·5°	178°	178°	177·5°	173°	178·5°	182°	177°
Te . .	113°	117°	105°	114°	111°	109°	115°	108°
t . .	103·5°	101°	95°	93·5°	89°	80·5°	87°	80°

Tb . .	178°	180·5°	180°	178°	175°	180°	180·5°	179·5°	mean	179°
Te .	114°	115°	114°	114·5°	116°	115°	115·5°	112°	,,	113°
t . .	108°	99°	94·5°	91·5°	93°	95°	95°	90°	,,	94°

"The columns represent readings at intervals of fifteen minutes.

"About 40 per cent. by volume was distilled off, giving benzine of 0·738 specific gravity with the following boiling-point :—

Up to 56°–60°	1·5	per cent.
,, 70°	5·5	,,
,, 80°	11·0	,,
,, 90°	19·5	,,
,, 100°	31·5	,,
,, 110°	44·5	,,
,, 120°	56·5	,,
,, 130°	68·0	,,
,, 140°	78·0	,,
,, 150°	85·5	,,
,, 158°	90·0	,,

"During the experiment there flowed off about 20 per cent. by volume of mixed oils of 0·915 specific gravity with the following boiling-point :—

Up to 179°–180°	0·5	per cent.
,, 190°	4·5	,,
,, 200°	6·5	,,
,, 210°	10·0	,,
,, 220°	13·5	,,
,, 230°	16·5	,,
,, 240°	19·5	,,
,, 250°	21·5	,, etc.

"In the still there remained about 60 per cent. by volume of mixed residue of 0·863 specific gravity and the following boiling-point :—

Up to	182°—190°	2·0 per cent.
,,	200°	4·5 ,,
,,	210°	7·0 ,,
,,	220°	10·0 ,,
,,	230°	14·0 ,,
,,	240°	17·6 ,,
,,	250°	20·5 ,, etc.

"4. With naked steam. For 100 volumes of distillate 30 volumes of condensed water from the steam. Tb at 150°, corresponding to about 5 atmospheres pressure :—

Tb	154°	151°	154°	155°	147·5°	148·5°	148·5°	150°
Te	98°	117·5°	110°	111°	113°	109°	108°	113°
t	100°	97·5°	96°	95°	97 5°	95·5°	95·5°	96°

Tb	150 5°	148°	146·5°	147 5°	150·5°	146°	150.5°	150°	mean 150°
Te	117°	112°	107°	110°	108°	104°	108°	110°	,, 109·5°
t	90°	94°	96°	95°	93·5°	93°	94°	94°	,, 95°

"The columns represent readings at intervals of fifteen minutes.

"About 42 per cent. was distilled off, giving a benzine of 0·744 specific gravity and of the following boiling-point :—

Up to	59·5°—70°	2·5 per cent.
,,	80°	7·0 ,,
,,	90°	16·5 ,,
,,	100°	27·5 ,,
,,	110°	40·0 ,,
,,	120°	52·0 ,,
,,	130°	64·0 ,,
,,	140°	74·5 ,,
,,	150°	83·0 ,,
,,	160°	88·5 ,,
,,	163°	90·0 ,,

"During the experiment there flowed off about 24 per cent. by volume of mixed oils of 0·916 specific gravity and the following boiling-point :—

Up to	190°—200°	2·5 per cent.
,,	210°	5·5 ,,
,,	220°	8·5 ,,
,,	230°	11·5 ,,
,,	240°	14·5 ,,
,,	250°	18·0 ,,

"In the still there remained about 32 per cent. by volume of mixed oil residue of 0·914 specific gravity and the following boiling-point :—

Up to 186°–190°	1·0 per cent.
,, 200°	4·0 ,,
,, 210°	6·5 ,,
,, 220°	9·5 ,,
,, 230°	13·5 ,,
,, 240°	16·5 ,,
,, 250°	19·5 ,, etc.

"Naked steam, therefore, as was to be expected, very importantly lowers the temperature, *t*, of distillation. An exit temperature of 94° in a mixture of 18 to 30 per cent. of steam to distillate is too high to retain a benzine boiling at 150°. The admixture of steam has a great influence, greater than the still temperature. The proportion of steam to distillate can be brought to any desired ratio by regulating Tb and Te to secure the required benzine. For every ratio of distillate to steam, *t* varies. If the steam proportion be reduced to nil, the temperature, *t*, must be raised to a maximum of 120°. For example, to obtain the ratio 18 per cent. of steam (experiment 3), *t* must be below 94°, about 85°, and Tb, Te must be lowered to 179° and 113° respectively.

"Per contra, if the pressure is diminished from 5 atmospheres (150° C.), steam must be used, but within the range of 30 per cent. of the distillate (experiment 4), giving *t* below 95°.

"Lastly, as regards the use of a vacuum in oil distillation if carried only to 70° (steam temperature), a vacuum is not unconditionally advantageous. If there has been already a decomposition, or quasi-cracking action, below 270°, (1) the proportion of kerosene may have diminished, (2) that of the substances removable by washing with sulphuric acid may have increased (and therewith the need of chemical treatment), and (3) the quality of the oil may have deteriorated by increased carbonisation : all of which would be remedied by the use of a vacuum. It may, however, be laid down definitely that decomposition, indicated by a gentler rise of the curve of specific gravities, commences at 270°. That is, a vacuum is not unconditionally necessary for the kerosene fraction, especially if the distillation is assisted by steam. If higher-boiling oils are to be carried over with the kerosene, the use of a vacuum is highly advisable."

With reference to the amount of heat dissipated through the coils of an ordinary condenser, and this may be taken as applying also to preheaters, Mr George E. Davis[1] states : "That for ordinary con-

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[1] *A Handbook of Chemical Engineering*, 1902, vol. ii. pp. 260, 261.

densers it is safer to take as a basis for designing 1000 B.T.U. per square foot per hour than to work upon the supposition that 250 B.T.U. will be dissipated per square foot per hour for each degree F. of mean difference between the internal and external temperature."

For the extraction of oils from seeds with petroleum ether (specific gravity 0·633), not less than 2 square feet of condensing surface should be provided for each gallon of distillate per hour with a condensing water supply of 3 gallons per gallon of distillate. The inlet temperature of the water is referred to as 50° F., rising 50° F. in its passage through the condenser. When the temperature is higher than 50° F., due to tropical or other conditions, ample allowance must be made, and the condensing surface increased accordingly.

Regarding condensers, Iltyd I. Redwood[1] states: "The area of the condensing surface of the condenser worm and the diameter of the coils largely affect the running of the still and the quality of the distillate. There should be sufficient outlets from the still to allow of the oil in the still being kept in contact with the heat for only the shorter length of time necessary to ensure a proper separation of the impurities intended to be removed by the distillation. The condensing capacity should be sufficient to allow of the still being run at minimum rate without causing loss of vapours due to imperfect condensation, and, in order to ensure this, there should be 1 square foot of condensing surface for every gallon of oil distilled per hour for heavy oils; 1¼ square feet for burning oils; and 1¾ to 2 square feet for naphtha or other very volatile oils.

"As regards the size of the condenser worm, the internal area of the cross-section of the worm at the inlet to the condenser should be 0·05 square inch per gallon of distillate per hour, or, if the distillate be run at the rate of, say, 200 gallons per hour, the size of the worm should be $\left(\sqrt{\frac{0{\cdot}05 \times 200}{0{\cdot}7854}} = \right)$3·102 inches in internal diameter. That size should be continued to 2½ inches for the next third and to 2 inches for the last third. . . .

"The quantity of water necessary to effect the condensation of the oil vapours and cool the distillate to any desired point can be calculated by the aid of the specific and latent heat figures given in the following table.

"For example, we will suppose that we have 1000 gallons of ·756 specific gravity naphtha to distil, and that the distillation will be conducted at the rate of 100 gallons per hour, then—

"1000 gallons at ·756 specific gravity = 7560 lbs.

"The latent heat of vaporisation of ·756 naphtha = 133·5° F.

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[1] *A Practical Treatise on Mineral Oils and their By-Products*, pp. 199, 201.

"Therefore (7560 × 133·5 =) 1,009,260 thermal units will have to be extracted from the vapours in order to condense them to fluid at 175° F. temperature. The condensed fluid (*i.e.* distillate) has to be cooled to 60° F., and as the specific heat of the distillate is 0·570 (water being 1·000), the amount of heat that will now have to be extracted will be 7560 × 175 − 60 × 0·5104 = 443,741 thermal units; therefore, total thermal units to be extracted will be 1,009,260 + 443,741 = 1,453,001. As the distillation takes ten hours, the number of thermal units per hour to be absorbed by the condenser will be 145,300. We will now suppose that the initial temperature of the condensing water is 50° F., and that the temperature of the overflow will be 80° F. As the specific heat of water is 1, and the temperature of the water will be raised 30°, each pound of water will therefore absorb 30 thermal units, and as 145,300 thermal units have to be absorbed (145,300 ÷ 30 =), 4849 lbs. (= 484·9 gallons) of water per hour will be required.

TABLE VII.—CONSTANTS OF PRODUCTS.

Name of oil or wax.	Specific gravity.	Boiling- or melting-point.	Latent heat of vaporisation.	Vapour density.	Specific heat.
		° F.	° F.		
Gasoline	·642	70	100·2	..	0·5800
Naphtha	·720	115	100·6	3·005	0·5687
"	·756	175	133·5	3·190	0·5704
Burning oil	·810	260	105·4	4·270	0·4991
Intermediate	·843	545	..	7·770	0·4950
Marine	·836	..	..	..	0·4561
Pressed oil	·885	..	..	..	0·4413
Lubricating oil	·865	..	..	..	0·4492
" "	·890	..	..	..	0·4441
Refined paraffin wax	..	81½	..	..	0·5584
" " "	..	118½	..	..	0·4164
" " "	..	120	..	..	0·4069
" " "	..	130	..	..	0·3659
Stearine	..	128	..	..	0·3529

"The above figures are based on the assumption that the boiling-point of naphtha would be 175° F. throughout the distillation, but this, of course, would not be the case, and more water than the above figures call for would be required. Also, a condenser is never perfect in its action owing to the worm becoming more or less covered with sediment, etc., and more water will be required again in this case; however, the approximate quantity of water required can be determined near enough for practical purposes by calculating as above and then adding 10 per cent."

CHAPTER V.

PARAFFIN EXTRACTION AND REFINING.

The heavy oil fractions, containing solid paraffins, which are separated from the crude oil in the continuous distillation plant are transferred from the receivers there to those at the paraffin shed, as the extraction plant is termed. Here any water they may still contain is allowed to subside, and if, as is frequently the case, they should contain carbonaceous matter in suspension, and which if allowed to pass on into the system will create trouble by clogging the filter cloths, it is filtered on its passage to the cooling apparatus. These are cooled artificially, and refrigerators working on the compression system are almost, if not entirely, employed in modern works.

The following diagram explanatory of the compression system of refrigeration is taken from T. B. Lightfoot's article, "Refrigeration and Ice Making Machinery." [1]

For those wishing to study the matter more fully, reference to the above article, to which a bibliography of works on the subject is appended, is recommended.

The following quotations therefrom are now made in explanation of the general principle of the working of refrigerators, without going too deeply into the whole subject :—

"There are three principal parts: refrigerator or evaporator, compressor, and condenser. The refrigerator usually consists of a coil or a series of coils connected at one end with the suction of the compressor, the delivery from the compressor being connected with the condenser, which also consists of a coil or series of coils. The refrigerator coils are surrounded with the substance to be cooled, and around the condenser coils cooling water is circulated. The bottom of the condenser coils is connected to the refrigerator coils by a pipe in which is a valve for regulating the quantity of liquid passing from the condenser to the refrigerator. The machine being in action, the refrigerating liquid flows into the refrigerator, in which the pressure

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[1] Thorpe's *Dictionary of Applied Chemistry*, 1913, vol. iv. p. 556.

is maintained so as to give the desired temperature of evaporation, and as this temperature is always below the temperature of the substance outside the coils, heat passes in from the outside and causes the liquid to evaporate at the temperature, T_1, the vapour being continually drawn off by the pump which compresses and delivers it into the condenser, T_2, which is somewhat above that of the cooling water. In the condenser heat passes from the compressed vapour into the water, the vapour being condensed, and the liquid which collects at the bottom returns through the regulating valve into the refrigerator."

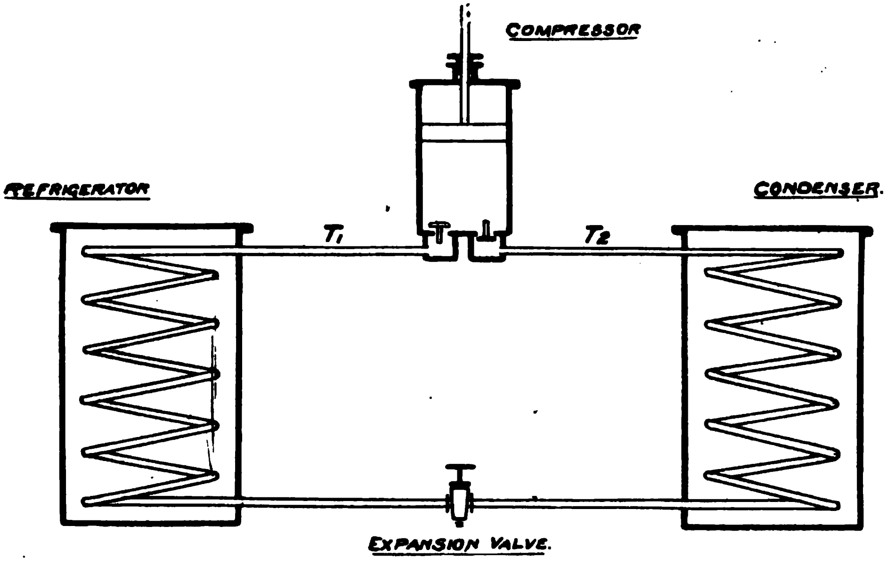


FIG. 72.—Refrigeration diagram.

In the above case the condenser coils are submerged, but better cooling effect is claimed for coils exposed to atmospheric influence with only a spray of water running over them. The cycle in the refrigerator is of course the same. Most large machines are now constructed with the atmospheric condenser.

The principal refrigerating agents in use are ammonia (NH_3) and carbon dioxide (CO_2). Which type of machine should be selected is a matter for expert opinion and advice, taking all the circumstances into consideration. Whichever type be decided upon may be either steam or electrically driven, and figs. 73 and 74 illustrate refrigerators operated by either form of power, and fig. 75 shows in plan and section a duplex steam-driven refrigerator arranged for brine circulation with submerged condensers.

There are three good arrangements for cooling heavy oil and paraffin, all designed by engineers prominent in the Scotch oil industry.

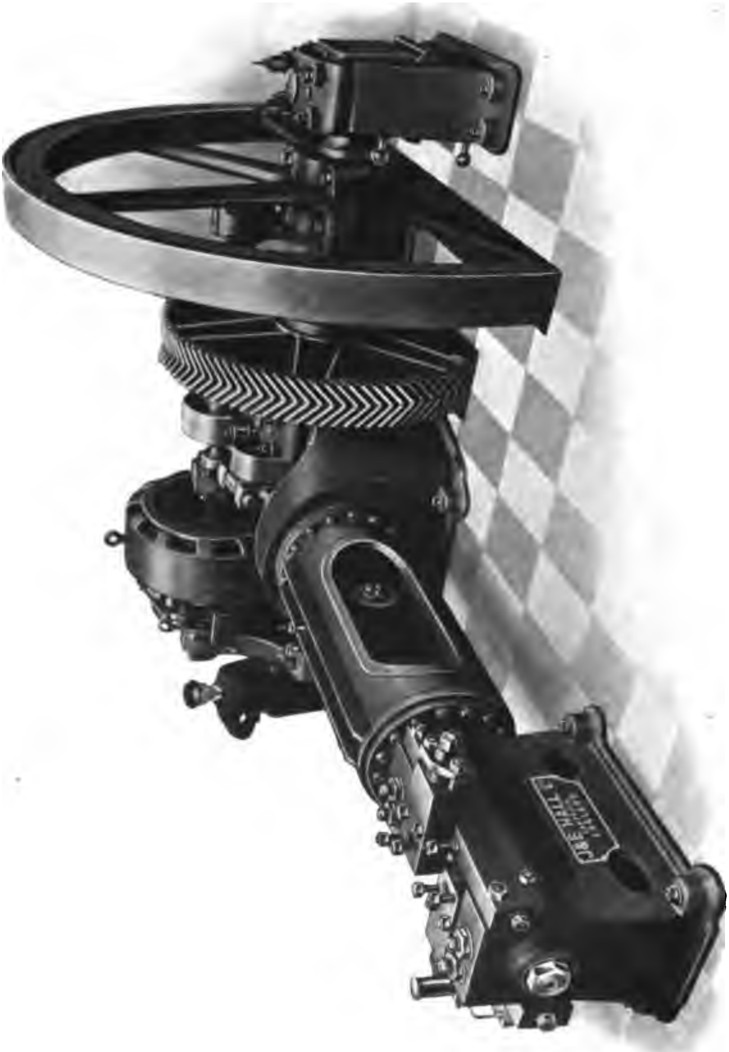
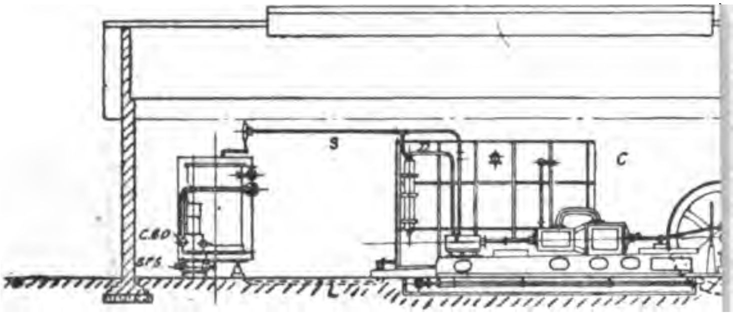
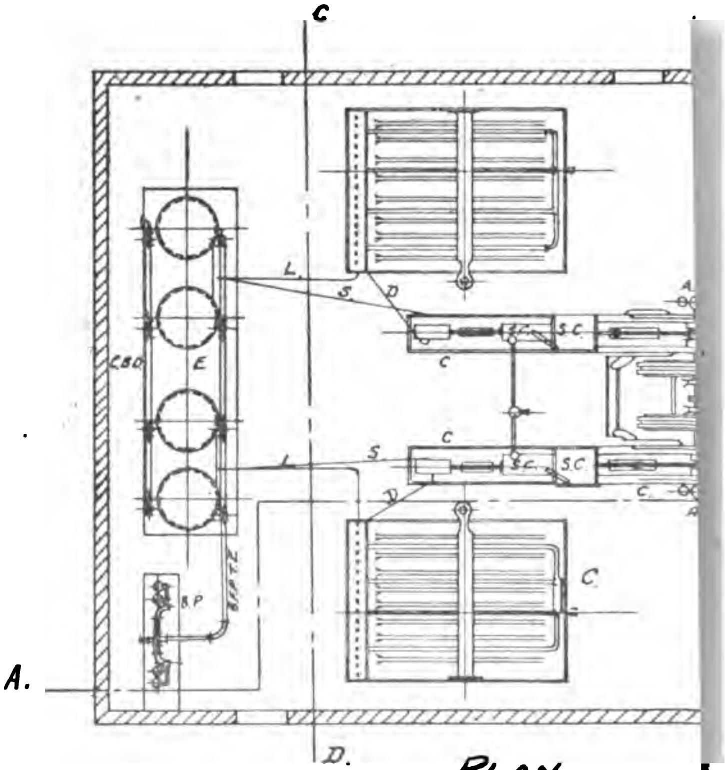


FIG. 73.—Electrically driven CO_2 refrigerator.

(1) The late Norman M. Henderson, Manager of the Broxburn Oil Co., Ltd. (fig. 76).



SECTION THROUGH A.B.



PLAN.

FIG. 75.—DUPLE

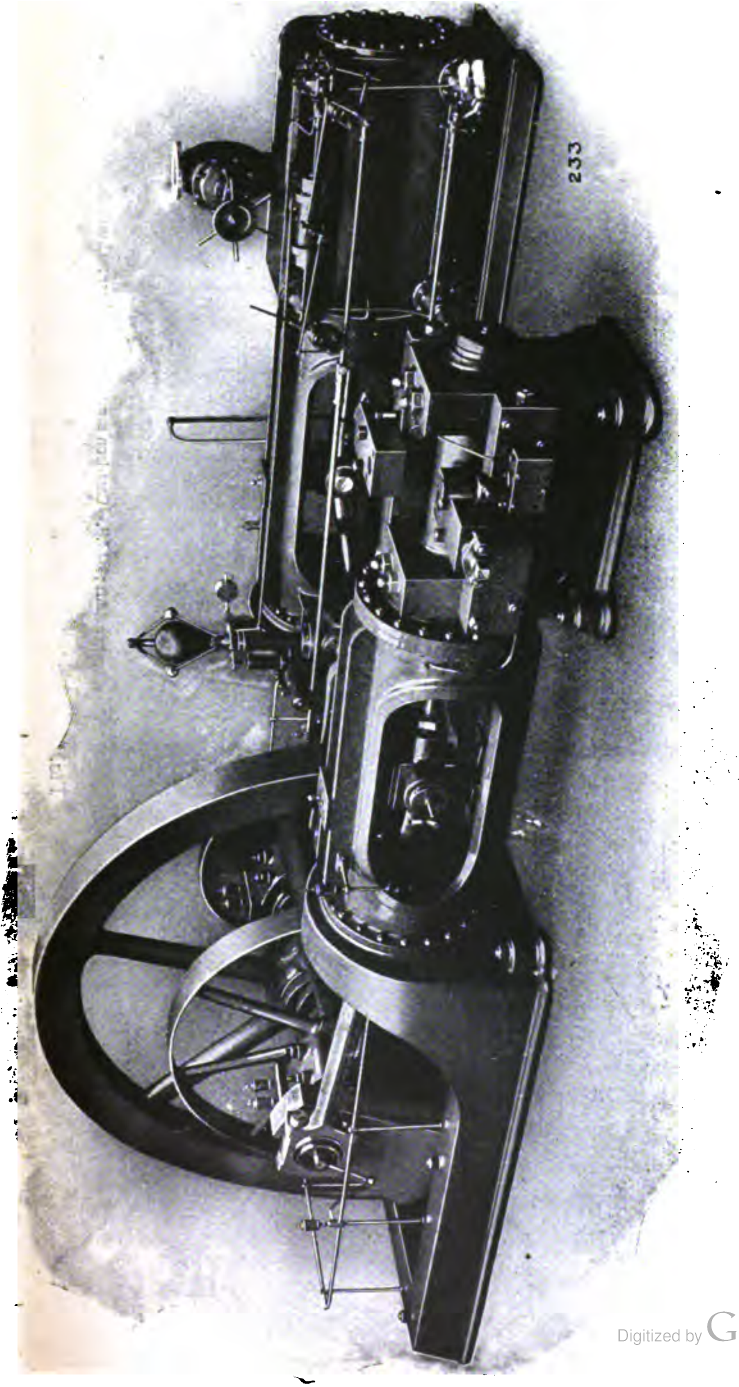
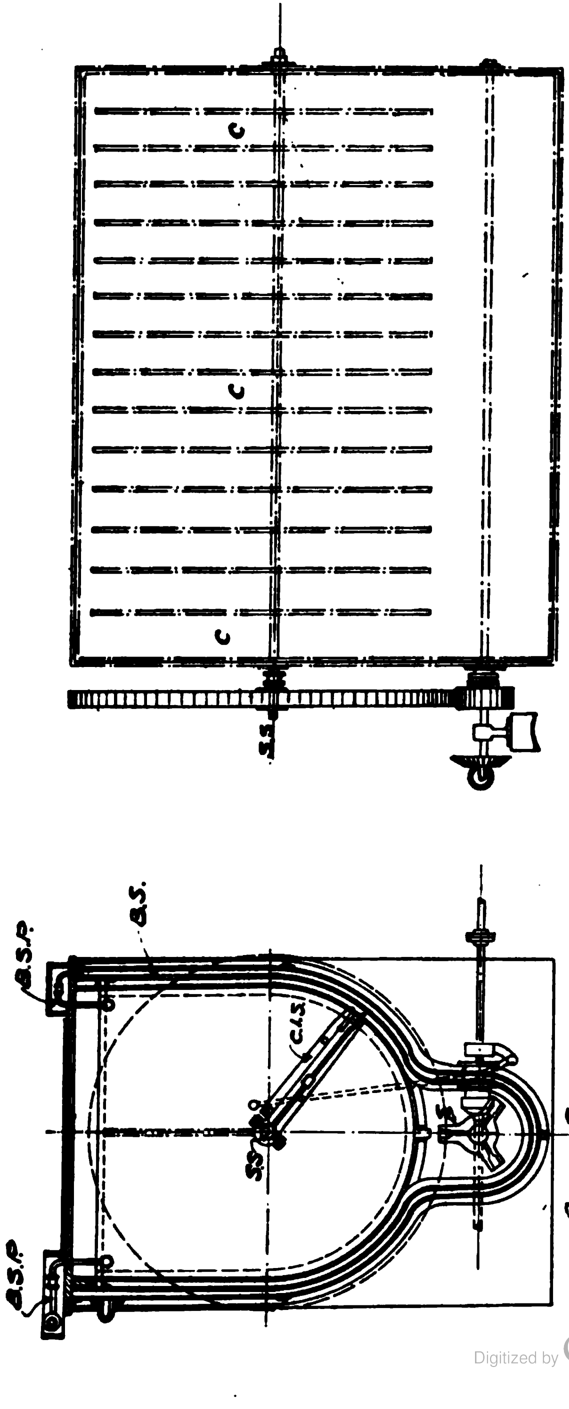


Fig. 74.—Steam-driven CO_2 refrigerator.



B.S.=Brine space; B.S.P.=Brine supply pipes; S.S.=Scraper shaft; C.I.S.=Cast-iron scraper; S.=Stirrer; C.=Cells.

Fig. 76.—Henderson cooler.

(2) James Bryson, Manager of the Pumpherston Oil Co., Ltd. (fig. 77); and

(3) A. C. Thomson, Manager of the Oakbank Oil Co., Ltd. (fig. 78).

All of these may be cooled by brine circulation, but the last-named is worked by the direct expansion of ammonia. In this case the cooler is the refrigerator in the example of refrigeration quoted above.

In the Henderson cooler, which has a capacity of 1000 gallons, the oil is contained in the inner shell, which is divided by a number of cells. The brine is pumped through these and overflows into the outer shell, finding its way to the return outlet through the brine space. The brine is in constant motion while the cooling proceeds, but as soon as the desired temperature is reached, it is shut off and the circulation only resumed when the cooled oil has been pumped away through the filter presses and the cooler recharged. The oil is kept in motion and prevented from solidifying on the surface of the cells by scrapers, revolved by a shaft as shown, and by a stirrer which prevents solidification taking place in the lower part of the vessel.

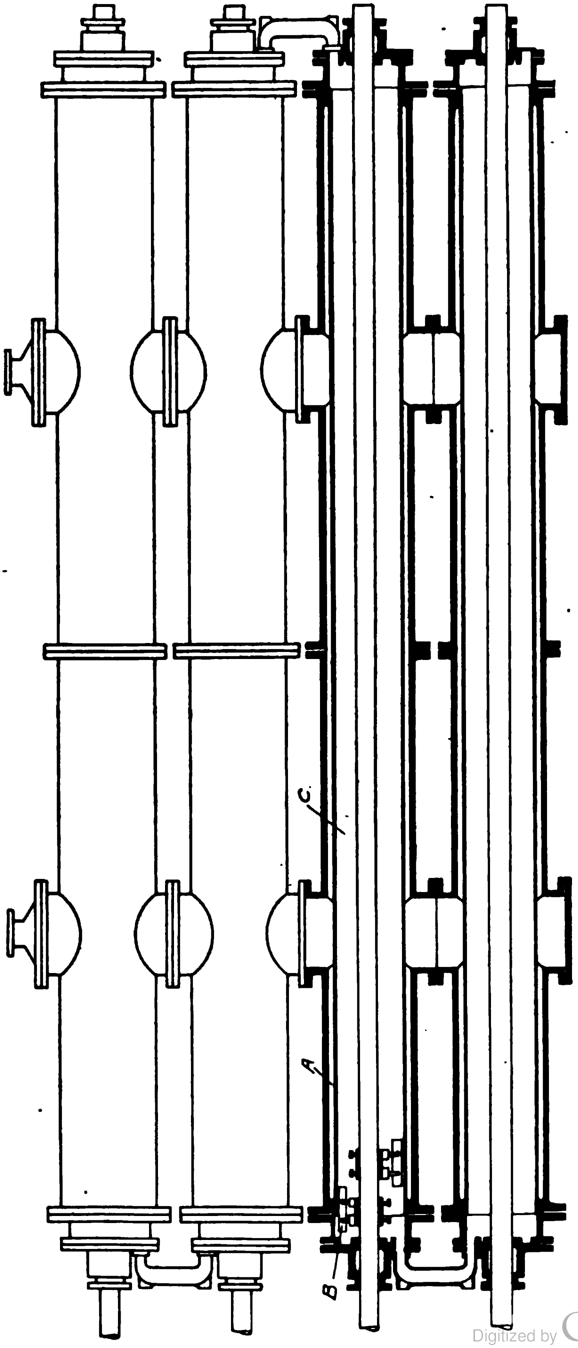
In the other two designs the oil and refrigerant pass in direct counter current, solidification being prevented by suitable scrapers.

The rate of cooling is regulated by the range of temperature through which the oil is to be cooled and the temperature of the brine or ammonia employed.

Whichever type of cooler be selected, a certain economy will result by using the cold oil passing from the filter plates to partially cool the oil entering the apparatus. This may be accomplished in a heat exchanger designed with ample surface to suit the temperatures at which it has been decided to work.

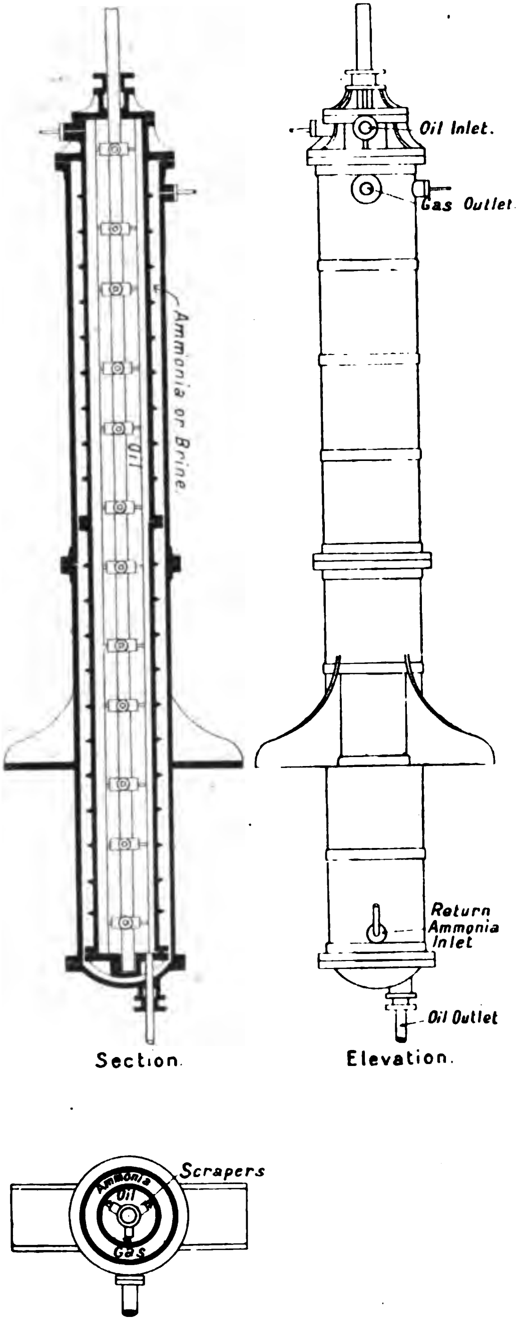
Cooling may be carried out in one or two stages, depending on the quantity of paraffin to be removed from the oil and the temperature at which the resultant filtrate is to be perfectly free from it. The second cooling is much slower than the first, because the temperatures are closer. For instance, in the first cooling the range may be from 80° F. at the inlet to 40° F. at the outlet, with brine leaving the refrigerator at 10° F. and returning at 15° F.; while in the second cooling the range will be from 40° F. to, say, 15° F., with brine at 0° F. returning at 5° F. The rate of filtration, too, will be slower in the second than in the first stage, because of the increase in viscosity due to the lower temperature.

The selection of a cooler will depend on the oil to be cooled, and for the first stage cooling of oil containing a large amount of high melting-point wax the author favours the Henderson apparatus. All three give good results, but as initial cost is an important factor in making the decision, it is better that each should be considered



A=Brine space; B=Scraper; C=Oil space

FIG. 77.—Bryson cooler.



Section.

Elevation.

Section.

FIG. 78.—Thomson cooler.

and the patentees consulted as to the efficiency of their invention under the conditions existing at the site on which the refinery is to be erected.

When the Henderson cooler is employed, the frozen oil is drawn from it by a pump and forced through filter presses to effect separation of the solid scale or crude wax from the oil; but when either the Thomson or Bryson cooler is employed, the oil leaving the preliminary cooler is forced through it right into the filter press, cooling taking place during the passage through the apparatus.

Fig. 79 illustrates the construction of a filter press, the plates of which are covered with a specially woven cloth strong enough to stand pressure. As the oil is pumped in, it separates, passing through the cloths, running into the troughs and thence to receivers placed at a lower level. These presses are sometimes closed by means of a hydraulic ram, as in the case of the illustration, to save hand labour. Fig. 80 is a drawing of a three-throw pump, which is the class of pump commonly employed for filling these filter presses. The scale fills up the space between the cloths, and when this is quite full and practically free from oil, the press is opened and emptied.

Filter press plates are made of cast-iron, ribbed on the surface and covered with perforated zinc sheet to support the cloth and prevent it sagging, or they are made on the "pyramid" principle, which offers a larger filtering area.

Pyramid Filter Presses—Recessed Plate Type Suitable for Oil Filtration.—The distinctive feature of the plates of this type is the raised rim which stands up above the drainage surface on both sides of the plate. When the plates are in position, a chamber is formed between every two plates, the depth of which is equal to twice the depth of the recess of the plates. A hole is provided in each plate through which the material to be filtered enters the chambers, and a filter cloth is hung over each plate having a hole in each half which corresponds to the feed hole of the plate, the joint round which is made by a clip. When for any reason it is desirable to avoid the use of cloth clips, then the cloth is made in two pieces sewn together round the hole; one half of the cloth is passed through the hole in the plate, and the two halves tied together over the top of the plate or held by suitable hooks. For oil work, however, the filter cloth is hung over each plate.

Fig. 81 shows a section through the head and a few chambers of a recessed type press with centre feed inlet. A, A_1, and A_2 are filter plates, forming chambers, B, B_1, and B_2, into which the material to be filtered enters by the inlet, D. The outlets for the filtrate connect to the ports marked E. The rims, C, of the plates are machined, so that when the plates are tightened up in position the cloths make

the joints between the plates quite tight. It will be seen that each chamber is thus completely lined with cloth, so that when the material to be filtered is forced in under pressure the solid particles are retained by the cloth, and the liquid portion freed from suspended matter and known as the filtrate, passes through to the outlet.

In this illustration three methods of fixing the cloths on the plates are shown: F and F_1 are screwed unions, G is an instantaneous clip which is fixed by a quarter turn, and H is one of the adjustable hooks to which the cloth is attached when sewn round the centre hole.

Pyramid Drainage Surface (fig. 82).—Actual tests have demonstrated that the "pyramid" drainage surface gives an increased rate of filtration of from 15 per cent. to 30 per cent. over that of the older forms.

The number of presses and filtering area required must be determined by experiment with the oil to be filtered. The amount of solid deposited and the rate of filtration will decide the number of plates required. After the scale is removed from the press it is dropped into a screw or other conveyor, which carries it to the hydraulic presses (fig. 83). It is here folded in strong cloth, also specially woven, and placed on baskets which lie on the shelves of the press. Pressure is then applied, and may reach as much as 20 to 30 cwts. per square inch, depending on the character of the scale. In this way the scale is freed from all but traces of the filtrate. On removal from the press, the cloths are opened and the dry scale transferred to a melting boiler, where it is melted with open steam, settled free from mechanical impurities, and blown in the liquid state to the next stage of the process—paraffin refining. Fig. 84 shows a well-arranged paraffin shed.

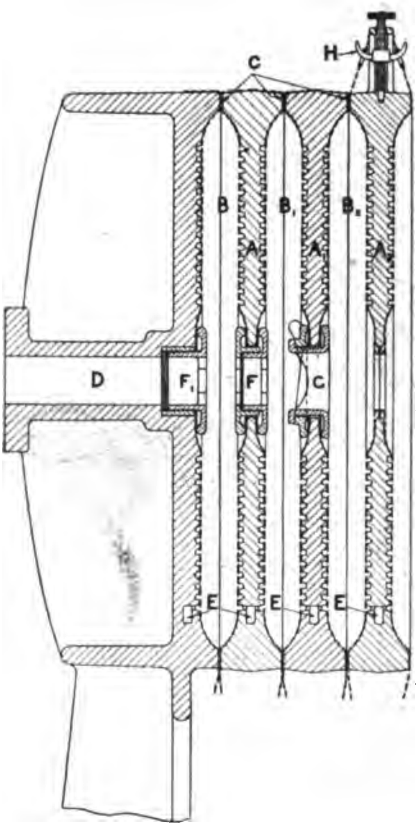


FIG. 81.—Pyramid filter press (section).

Before passing to that section, attention is drawn to another process for the separation of paraffin from paraffin oil mixtures, perhaps worthy of attention, patented on the Continent by the Triester Mineralöl Raffinerie, Trieste, and introduced to notice in this country by Mr Von Groeling. It is, to quote a translation of the official description, "based on a quality of paraffin not hitherto known in the industry, *i.e.* that under certain conditions it precipitates out of the oily solution. These conditions are that a fluid mixture of paraffin and oil is cooled down uniformly through and through without being exposed to the usual motion in cooling apparatus, it being left almost at rest." What slight motion is required to

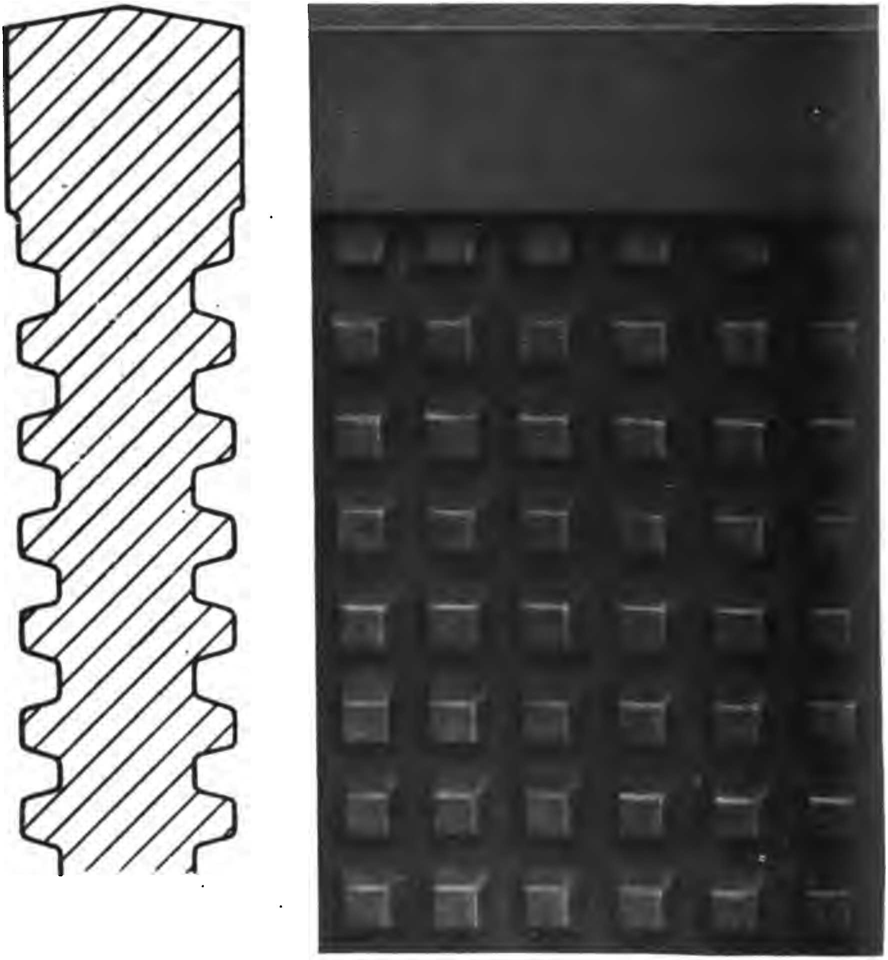


FIG. 82.—Pyramid filter press (drainage surface).

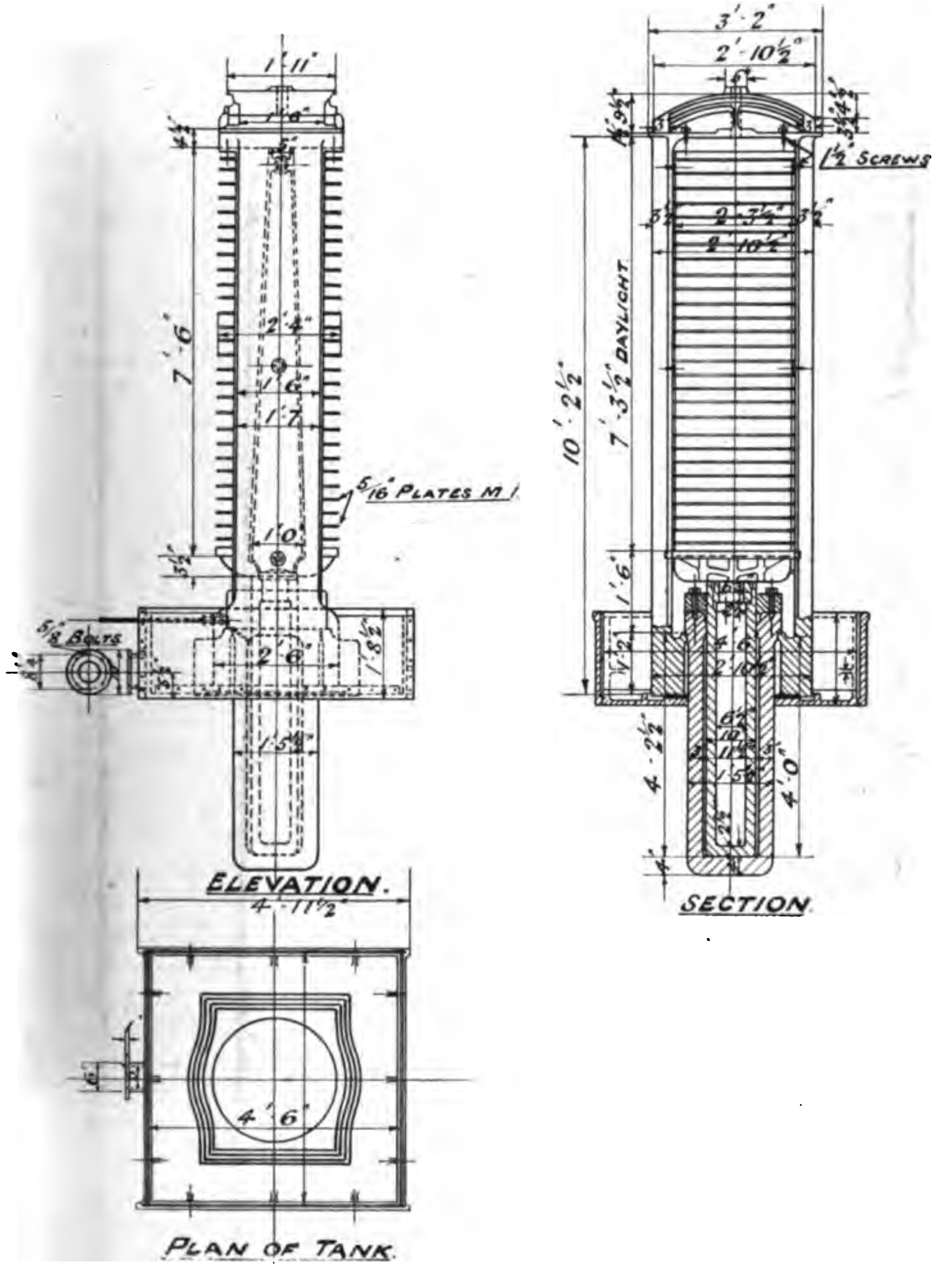


Fig. 83.—Hydraulic press.

prevent the liquid next the casing of the vessel from solidifying, and forming a non-conducting coating, is provided by the slow movement of a skeleton scraper round the inner surface of the vessel, which is simply cooled by a stream of water running down the outside. "The precipitation of the paraffin crystals starts at the moment when the setting-point is reached in the cooling described, and this is allowed to continue until a little more than the lower half of the cylinder is filled with paraffin crystals." The cylinder, or tank, in which the operation takes place is fitted with a false bottom of gauze, on which the paraffin precipitates. The oil is then run off, which it does freely, and may then be cooled artificially to remove the softer paraffins, while the precipitated paraffin goes to the refining plant. The apparatus is exceedingly simple, consisting of a water-cooled tank, and a slowly rotating scraper or paddle (figs. 85 and 86).

The crude scale, even after hydraulic pressing, still contains traces of the heavy oil, and this must be removed before the colour becomes perfectly white. To attain this object, and to fractionate the scale into one or more grades of wax, it is subjected to the "sweating" process. This may be performed separately on each grade of scale, or together, as found by experiment to be the better procedure. The original sweating process was brought out many years ago by Price's Candle Co., and was worked as follows, and the same arrangement of trays is in daily use in many refineries for forming the finished wax into blocks of convenient size to pack for transport, the blocks being despatched in wooden cases or in bags, the latter usually double :—

The melted scale, freed from dirt and water, was run into a wooden trough set on the top of an iron rack supporting twelve dozen flat trays, each containing 10 lbs. of wax, in twelve vertical rows. An outlet, stopped with a plug, over each row, allowed the melted scale to flow into the uppermost tray, from which it overflowed through a slit in the end to the tray underneath, and so on to the twelfth or bottom tray. The racks were arranged in a well-ventilated apartment. After cooling to atmospheric temperature, the solidified cakes were loaded on a trolly, and conveyed to the sweating room. There they were laid on shelves tilted at a slight angle to permit the sweatings to reach the collecting troughs quickly. To ensure as good a separation of the oil as possible, the shelves were covered with cocoa-nut matting.

As will be obvious, the process entailed a large amount of labour, and here again Mr Henderson showed his genius, inventing in 1887 what is universally known as "Henderson's sweating stove." This consists of a frame on which are set, one above the other, nine trays measuring 21 feet long by 6 feet broad and 6 inches deep and having

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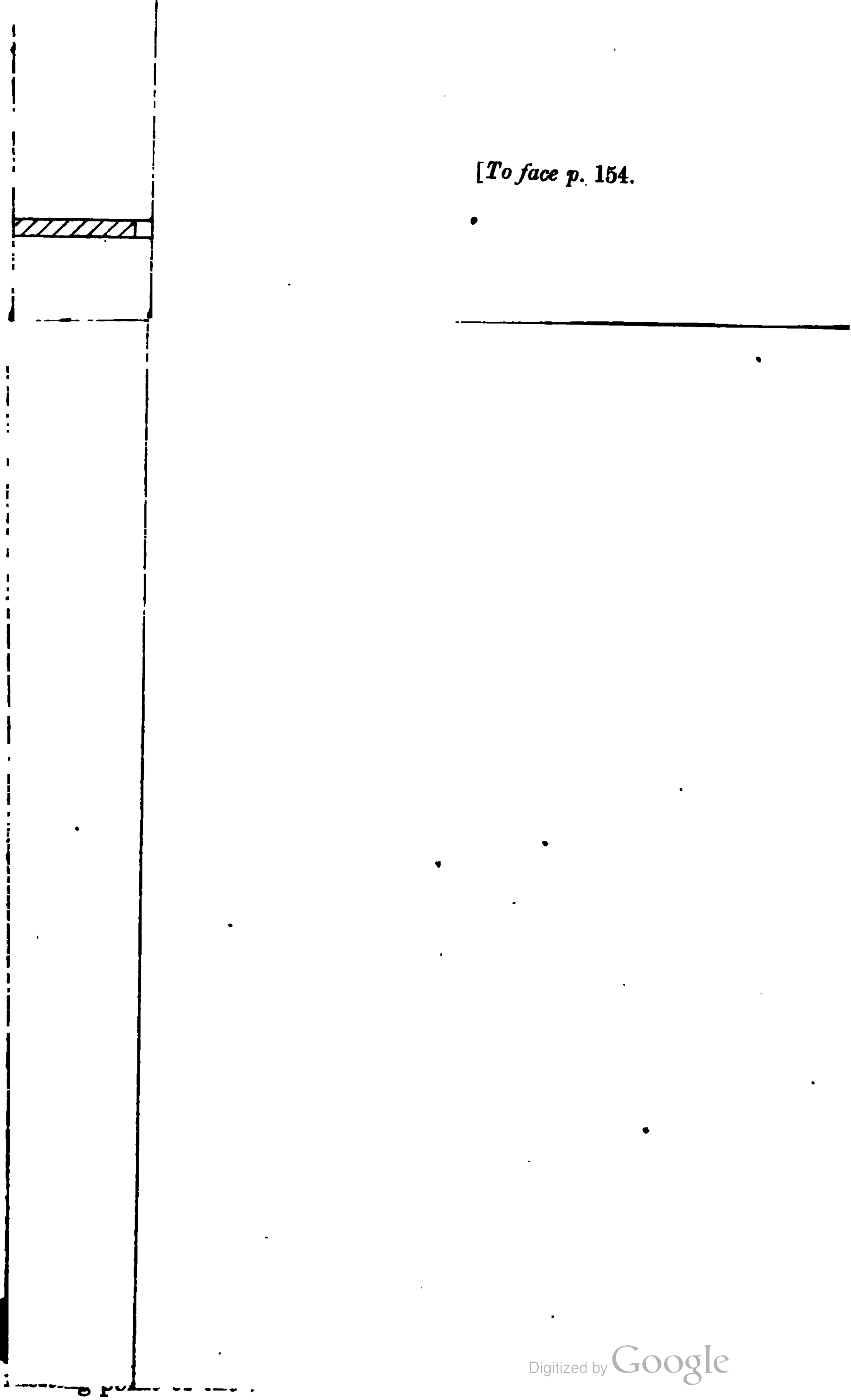

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a total scale capacity of 7 tons. Gauze is stretched across these, about 2 inches from the bottom, and the frame is provided with an arrangement for tilting the trays to an angle sufficient to ensure good drainage. The frames are erected, either singly or in pairs, in rooms heated and ventilated in turns. When in the level position the trays are filled with water as far as to the gauze. The melted wax is then run in, and the whole allowed to cool, ventilation being assisted if necessary with electric fans. The time required for this depends on the melting-point of the mixture to be sweated, and on the weather

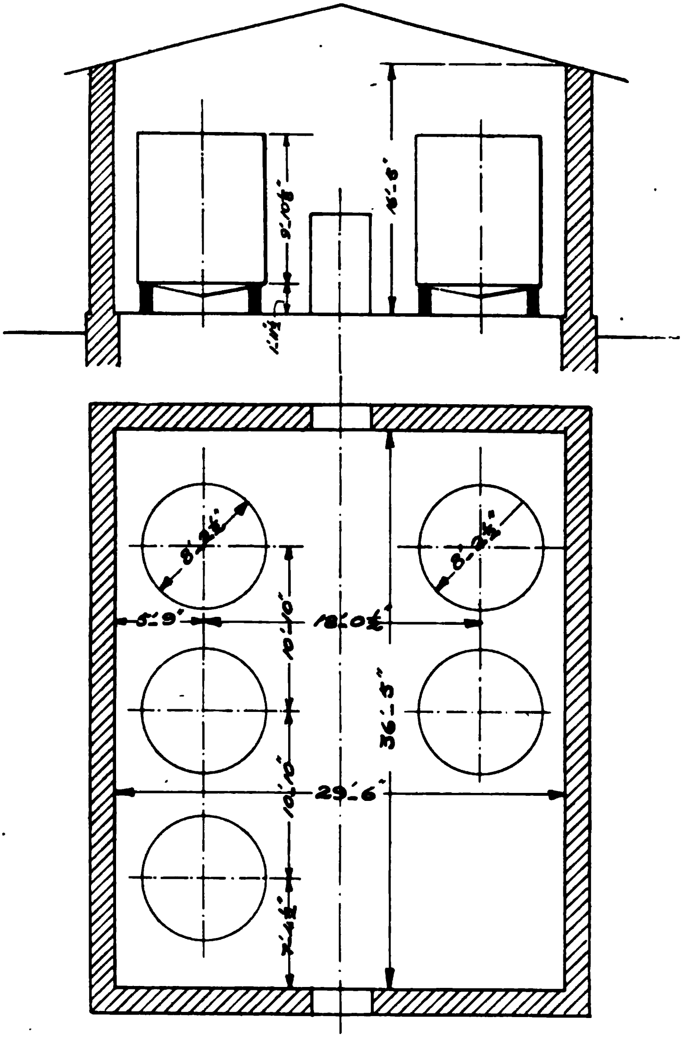


FIG. 85.—Paraffin crystallising plant.

conditions. When uniform solidification is reached, the water is run off, and the trays tilted. Heat is then applied, and sweating carried on until the residue is of the melting-point aimed at. The sweatings running off are fractionated to suit the general process. All the

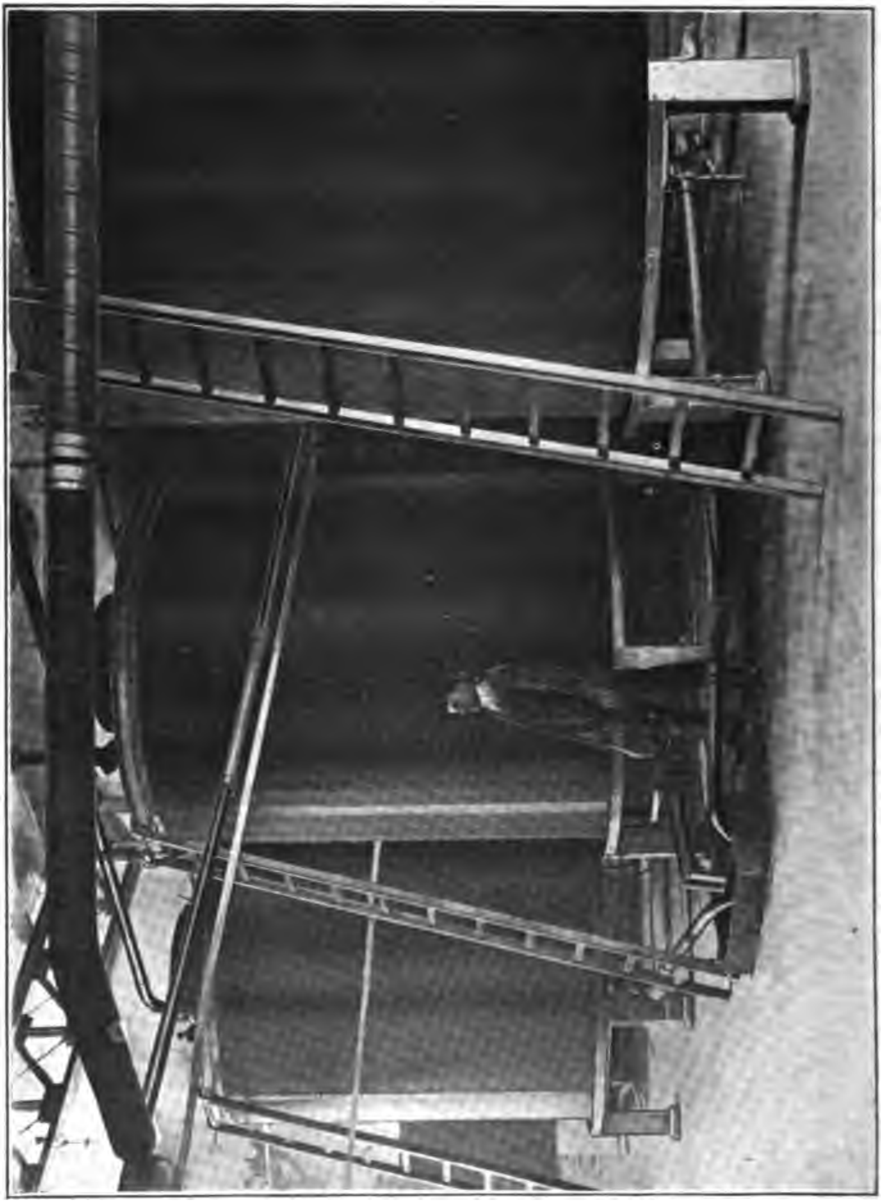
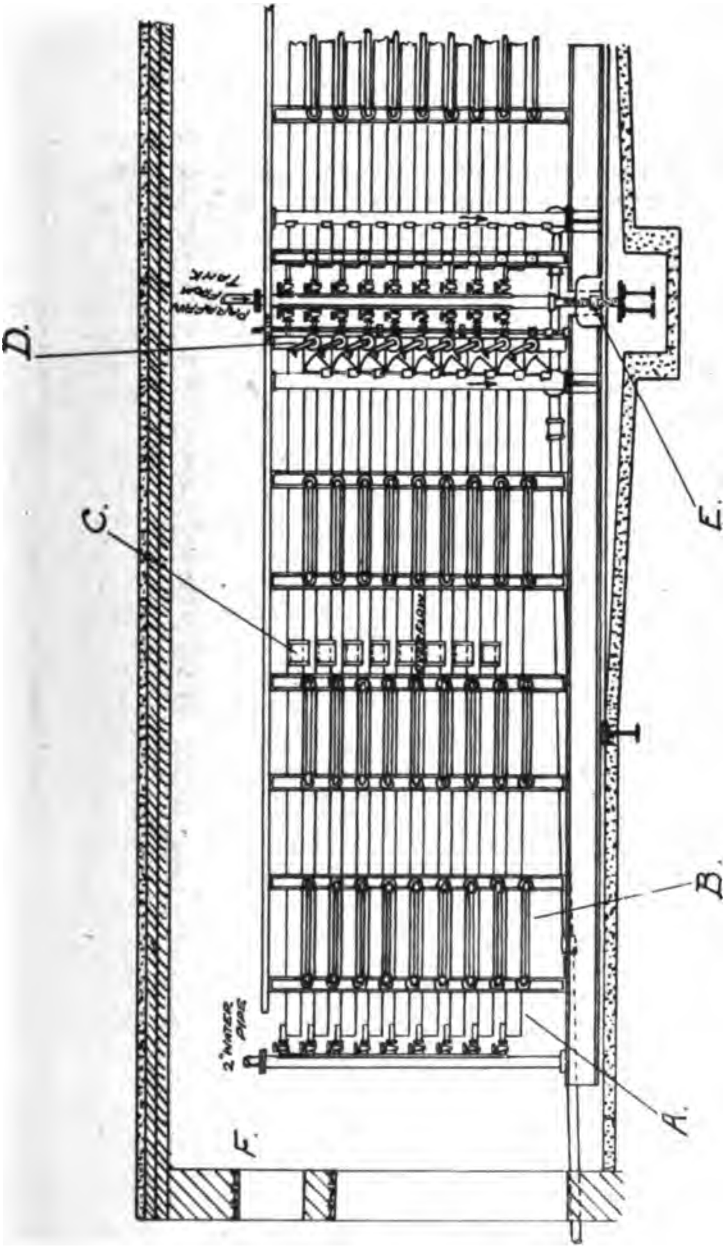


FIG. 86.—Trieste system of oil cooling.

sweats are thoroughly drained from the trays, and the residue on the gauze, being melted by steam, is diverted to its proper receiver.



A=Trays fitted with false bottom of wire gauze; B=Heating coils; C=Overflow; D=Swivel sweatings run off; E=Tilting screw; F=Sweating chamber fitted with heating coils on walls.

FIG. 87.—Henderson's sweating stove—tray form.

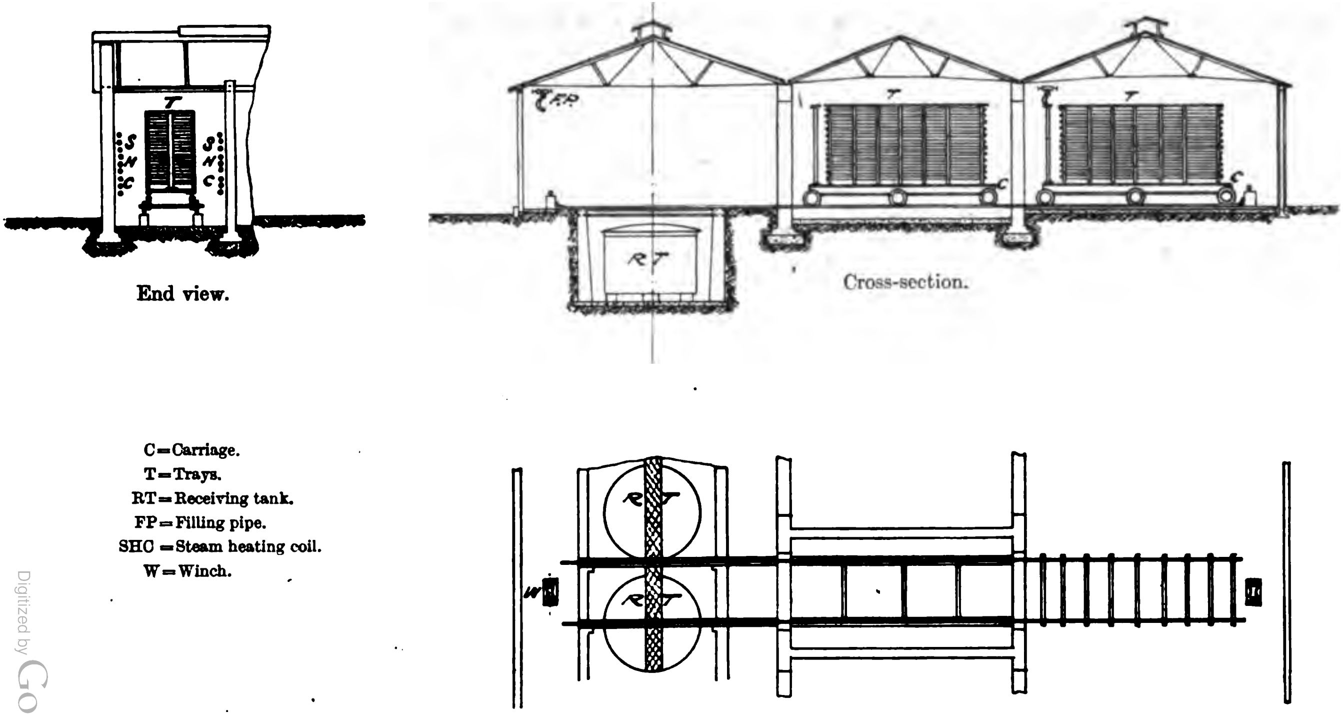
The frame is returned to its level position, and is ready for refilling with crude material (fig. 87).

In working this plant in a tropical country, trouble was experienced in cooling the wax contents of the trays uniformly. When the sweating chamber became heated with the steam coils, it was impossible to cool it and the trays to the proper point, without a considerable loss of time, because of the slight difference between their temperature when newly filled with scale and that of the atmosphere. To overcome this, in one refinery, two frames were fixed on wheels and arranged to be pulled out of, or into, the sweating chamber at will, a capstan being used for this purpose. A frame, while outside, and therefore removed from the influence of the heated building, is charged in the ordinary way. After cooling it is pulled into the chamber, and sweated, and on completion of that operation is pulled out into the open air. A second frame alternates, one frame entering the chamber every twenty-four hours. Working under tropical conditions, the capacity of the plant was increased 50 per cent. (fig. 88).

Mr Henderson also invented another form of sweating plant which occupies less space than the tray form, *i.e.* more scale may be sweated in the same cubic capacity (fig. 89). This, which is said to give excellent results, consists of upright cylinders, made of light sheet-iron, 9 feet high and 17 inches in diameter, within which other cylinders 7 inches in diameter are fixed. The latter are opened at top and bottom, and the annular space between them and the larger cylinders is fitted with a false bottom of gauze, a few inches above the real bottom, where the outlet cock is placed. The inner cylinder is covered, on the side next the outer cylinder, with several plies of wire gauze. The manipulation is the same as with the trays, the scale occupying the space between the two cylinders. The wire gauze acts as a wick to carry off the sweats as they part from the scale, and drainage may be assisted by the suction of a steam injector. In another arrangement of this system the cylinders are placed in an iron tank instead of a room, and are cooled by water in place of air circulation.

Another form of plant for sweating paraffin is that patented by Dr Pyzel, and "consists of a long passage provided with rails or tramways on which the sweating vessels placed on trucks or cars are run into the oven." The heat is supplied by blowing air over a hot coil into the passage so that the sweating takes place progressively as the trucks advance towards the outlet. An air-sluice arranged at each end of the tunnel ensures a minimum loss of heat when the vessels are entered or removed. The bottom of the passage slopes towards the outlet to facilitate the movement of the trucks, and the sweats are conducted into gutters arranged in the floor. The finished wax is melted in the outlet air-sluice (fig. 90).

Crude scale may be transferred from the paraffin shed to the wax



End view.

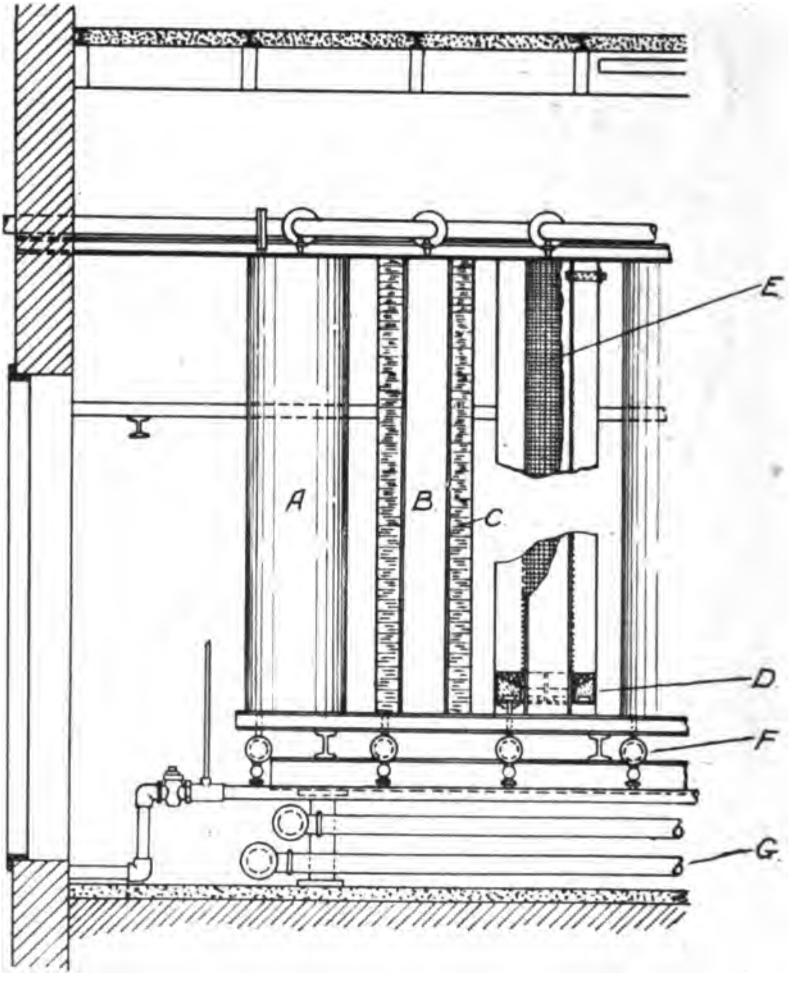
Cross-section.

Plan.

C = Carriage.
T = Trays.
RT = Receiving tank.
FP = Filling pipe.
SHC = Steam heating coil.
W = Winch.

FIG. 88.—Campbell's "pull-out" sweating stoves.

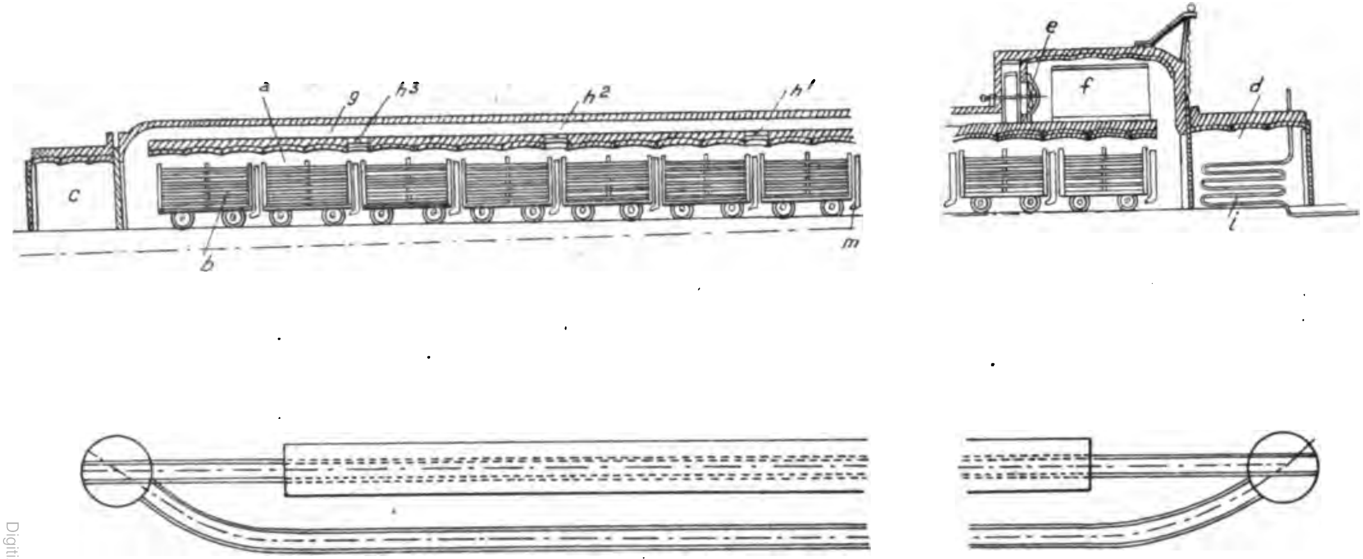
refinery in the solid state, but where possible, it is more convenient to do this in the liquid condition by blowing it through pipes. These require steaming before and after each batch is sent through, to prevent clogging.



A = Outer cylinder; B = Inner cylinder; C = Wax space; D = False bottom and water space; E = Gauze; F = Run-off pipes; G = Heating coils.

FIG. 89.—Henderson's sweating stove (cylindrical form).

The "sweats" are returned for redistillation, and the sweated wax (fig. 91), now of the desired melting-point, in one or more grades, passes through its final stage of refinement and decolorisation. Notwithstanding the treatment it has undergone, it still contains traces of colouring matter, which must be removed before it is



a=Sloping passage with rails; *b*=Sixteen trucks; *c* and *d*=Air-sluices; *e*=Fan; *f*=Heating coil; *g*=Air passage; *h*=Air dampers; *i*=Steam coil; *m*=Sweats pipe.

FIG. 90.—Pyzel's sweating plant.

suitable for candle manufacture. With this intention it is mixed with or filtered through some decolorising medium. Charcoal, fuller's earth, Japanese acid clay, and infusorial earth have been employed with varying results. Bauxite—a British patent, No. 16,617/1908, was granted to Dr J. J. Hood and A. Gordon Salamon for the treatment of mineral oils with bauxite in 1908, and it is owned and worked by the Oil Refining Improvements Co., Ltd., 175 West George Street, Glasgow—has proved very efficacious, not only for this purpose, but for the decolorisation of kerosenes and lubricants, and it has also proved valuable in absorbing odoriferous bodies which other treatments failed to remove from distillates. The wax is best brought in contact with the bauxite by filtration. Before use, and after grinding to a suitable degree of fineness, it is incinerated until all, or nearly all, the water in combination which it contains is expelled, and to obtain a good effect, all free moisture must be eliminated from both wax and bauxite. Filtration is continued until the average filtrate possesses the intended degree of colour, and this may be accurately controlled by the use of a Lovibond tintometer adapted

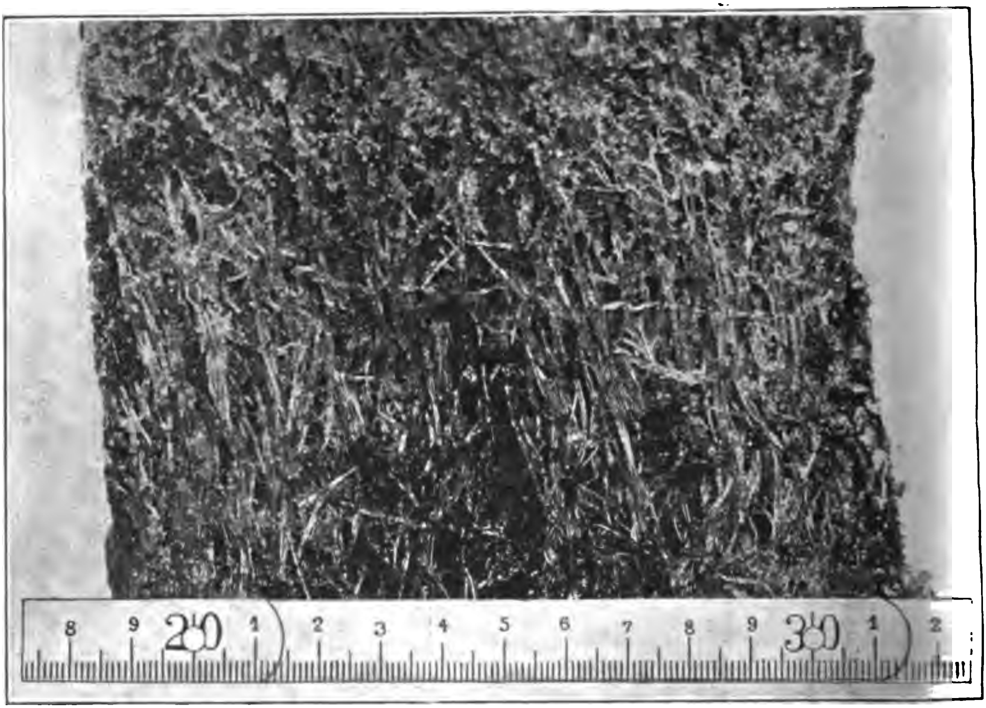


Fig. 91.—Sweated paraffin.

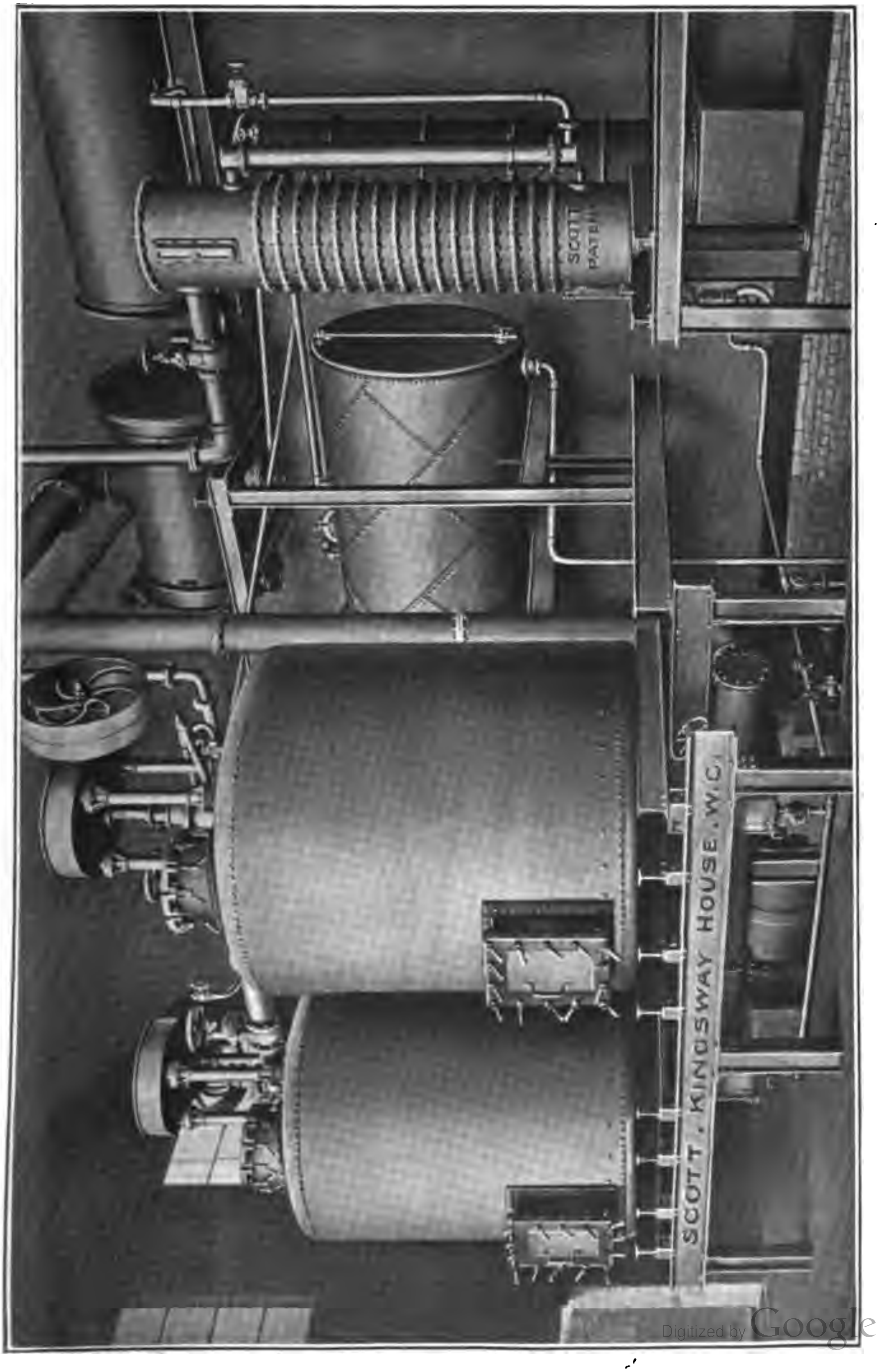


FIG. 92.—Scott extractor.

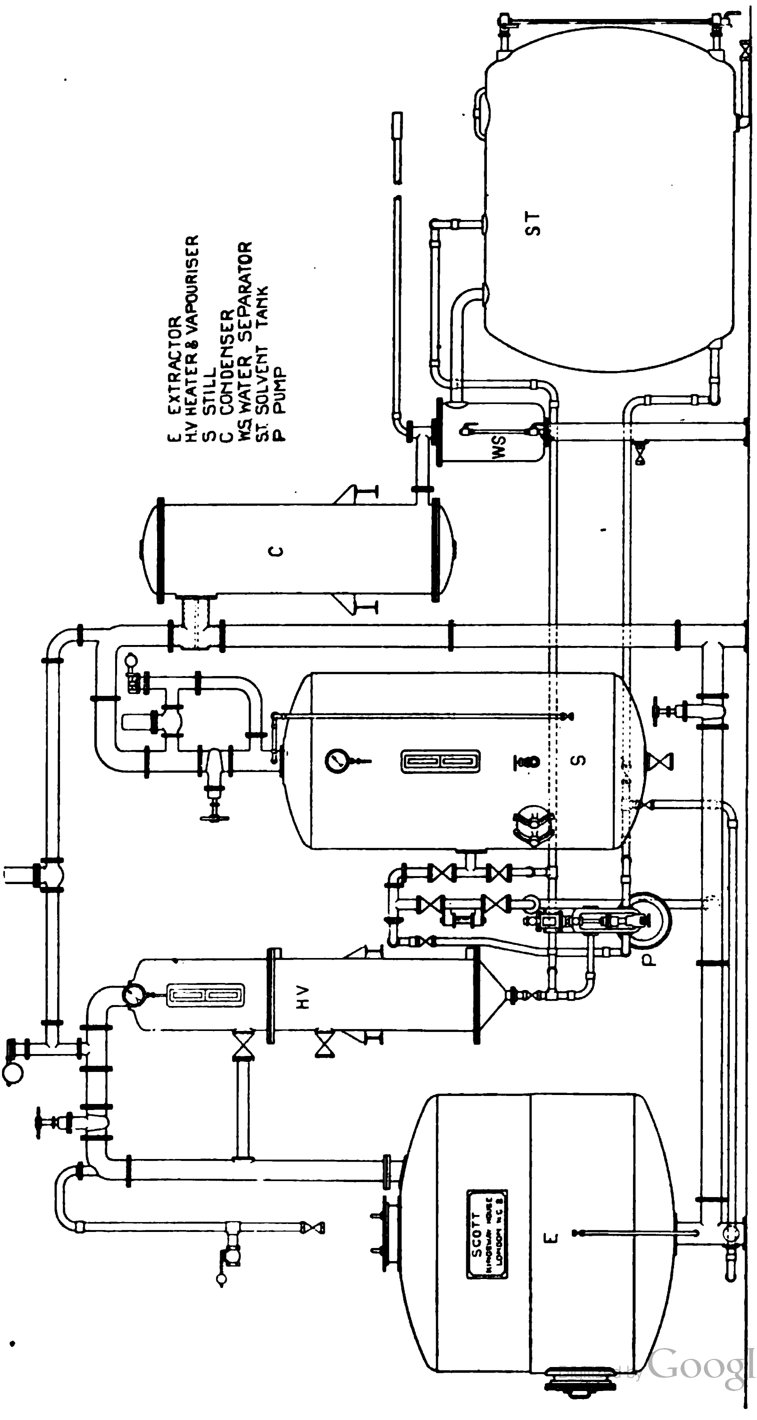
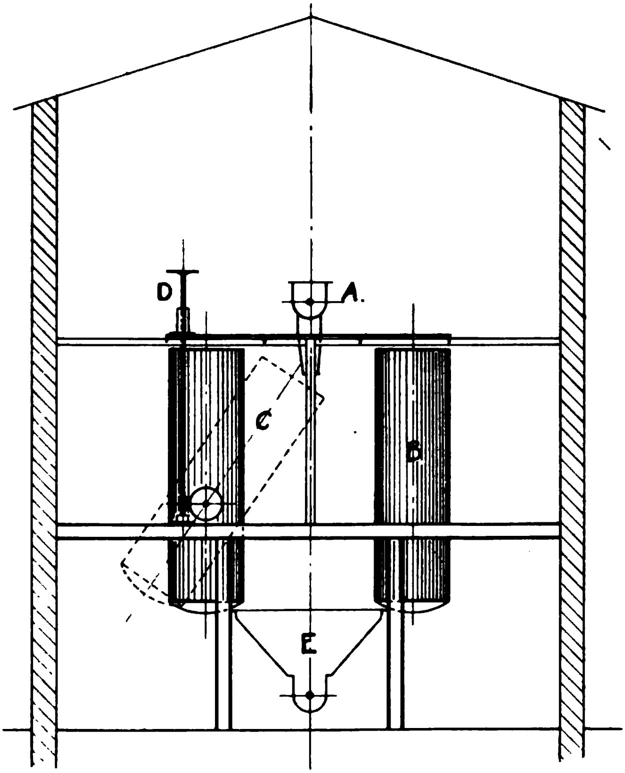


Fig. 93.—Scott extractor.

for the examination of melted wax, as already explained in Chap. I. For large manufacturers a filter containing 1 ton is a convenient size, as it permits 25 to 50 tons of wax to be filtered at each charge of bauxite. A filter of this type should be made without any seams, except for the run-off at the bottom. When the material is exhausted, it is discharged into a trolly for conveyance to an extractor, where the wax still adhering is dissolved out with petrol. A good type of extractor, which is particularly efficient for the purpose, is that designed by George Scott & Son (London), Ltd., and illustrated by figs. 92 and 93. A tilting arrangement inverts the filter while still hot, causing the charge to slide into the trolly (fig. 94). After the solvent is thoroughly expelled by steaming, the bauxite is conveyed to the incinerating furnace, where any remaining traces of hydrocarbon are burnt off, leaving it ready for use once more,

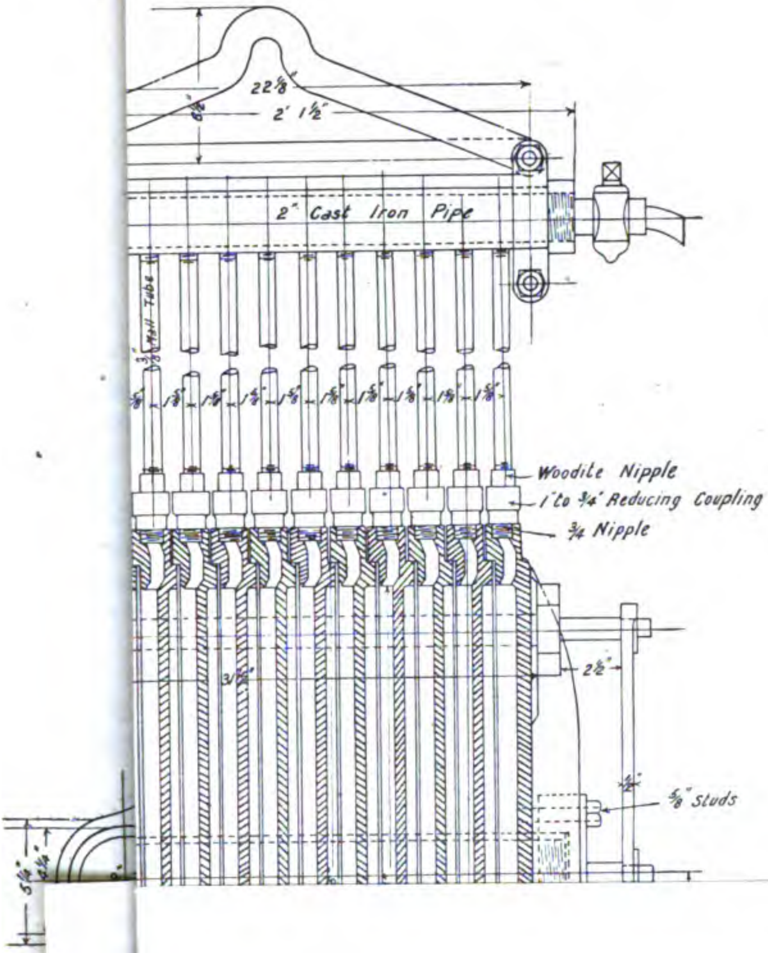


A=Conveyor for fresh bauxite; B=Bauxite filter; C=Bauxite filter tilted to receive charge; D=Tilting arrangement; E=Hopper or trolly for exhausted bauxite.

FIG. 94.—Bauxite wax filter.

and practically in possession of its original decolorising efficiency. A rotary furnace is very suitable for this purpose. Such losses as are sustained are chiefly mechanical, and are due to the lightness of the material.

To prevent traces of the filtering material reaching the finished blocks of wax it is, after decolorisation, passed through filter paper on its way to the cooling racks already described. The Bryson filter is an efficient apparatus for the purpose, and has to a great extent superseded the rather cumbersome cylindrical filter used for so many years (fig. 95).



certai

regulated by the melting-point of the paraffin and by the climate

CHAPTER VI.

CANDLE MANUFACTURE.

THIS involves the establishment of a complete department for the purpose. The building will be divided into rooms for melting tubs and pans, moulding machines, packing and boxing. Accommodation for storing supplies of materials will also be required, as considerable quantities of tissue paper and wrapping papers, labels and sealing-wax are used for making up the packets in which the candles are sold. Timber forms a large item among supplies, and is usually purchased ready cut in sizes convenient to make the boxes, which vary in dimensions according to the size of candle and number of packets to be sold. Timber so cut is termed "shooks." Those forming the sides of the box are stamped or branded with the market name of the candle and the name of the manufacturer. The shooks are fastened together with wire nails driven in by box-nailing machinery (fig. 96).

A is the hopper of the automatic nail feeder, and B the tracks down which nails run, and which are adjustable to different size and gauge of nails. C is the automatic cut-off feeder, and D the tubes down which the nails drop into the chucks, E, which carry the hammers and where they are held in vertical position until driven into the wood. As soon as this set of nails is driven, another lot fall automatically from the feeder into the chucks. The table, F, can be adjusted to suit the size of box, and on this the ends are nailed to the sides and the body of the box formed. G is a fence or guide, and H the operating foot lever.

The machine is adjustable for nailing on bottoms to the bodies. The machine illustrated is driven by the motor, J, and will turn out 500 to 600 boxes in ten hours.

Paraffin wax becomes plastic under the influence of gentle heat, the lower its melting-point the more readily is it affected. Candles made from paraffin therefore "bend" easily, and to prevent this a certain percentage of stearine is added. The amount necessary is regulated by the melting-point of the paraffin and by the climate in which the candle is to be burned. The addition of stearine also imparts a "skin" to the finished candle—paraffin wax alone being apt to stick to the moulds. A good grade of stearine should be used, for, if it contains much oleic acid, the candle made from it will, on

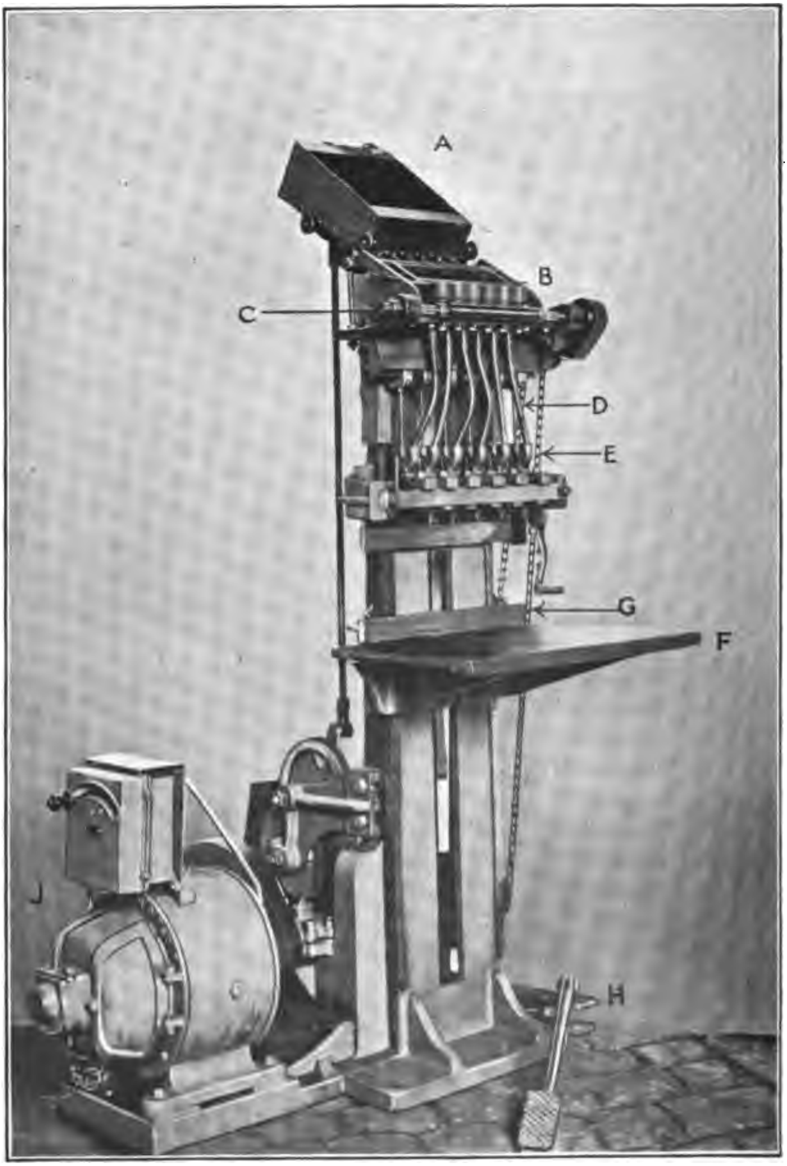
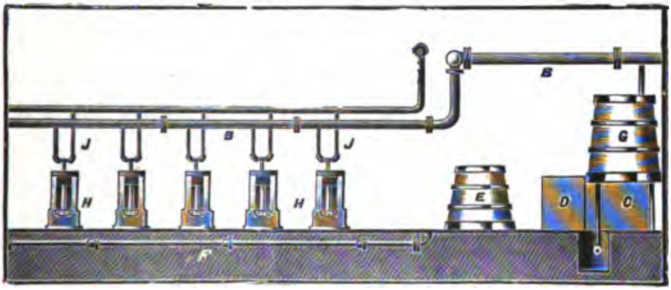


Fig. 96.—Box-nailing machine.

being kept for a time, develop an unpleasant rancid smell and discoloration.

The bending-point of a candle is determined by setting it in a perfectly perpendicular position in an air-bath with a glass door.



Plan and elevation.

A = Main steam pipe.
B = Main water pipe.
C = Separators.
D = Jacket pans.
E = Dirty tub.
F = Drain pipe.
G = Boiling-down vats.
H = Candle machines.
J = Inlet steam and water pipes and valves to machines.

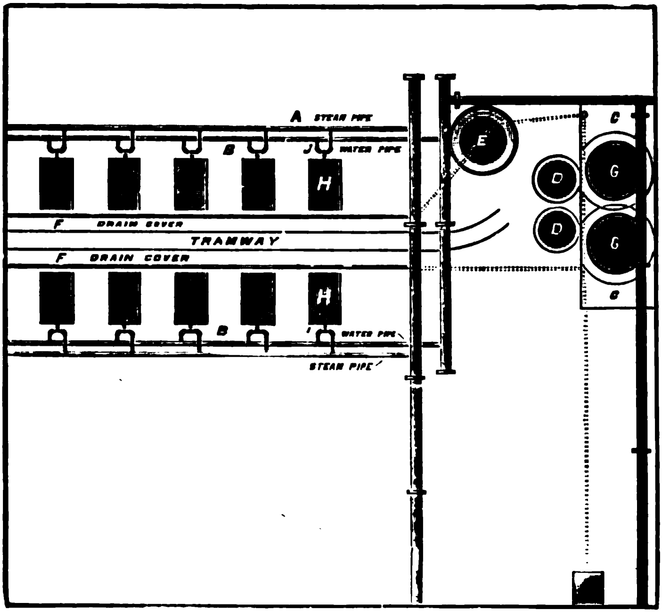


FIG. 97.—Plan of candle machine room.

The temperature is then raised 5° F. every half-hour until the candle bends off the perpendicular. A number of candles may be tested in this way at the same time.

Paraffin wax has a greater illuminating power than stearine, therefore the larger the percentage of the latter added to the former, the lower the illuminating value of the composite candle.

Candles may be made of any desired shade or colour by the addition of aniline dyes.

Wicks are purchased from the cotton spinners, plaited into such number of threads and strands as are determined to be most suitable for consuming the material of a candle of certain diameter at an approximate rate of burning. These are chemically treated by the makers, to ensure that they will be "self snuffing" when burned. Opinions differ as to the best chemical to use for this purpose, but ammonium nitrate, borax, ammonium phosphate, potassium chloride and chlorate have all been employed.

When the wicks are "pickled," or treated, by the makers, they may be reeled on paper bobbins, saving much time and labour in performing those operations at the candle factory. When wicks are purchased untreated, machines will be required for the purpose, and much care must be exercised in drying the wicks after impregnation and reeling them into bobbins of suitable size to suit the moulding machines. The arrangement of moulding machines, melting pans, and all the requisite connections for steam, water, and drainage are illustrated by fig. 97.

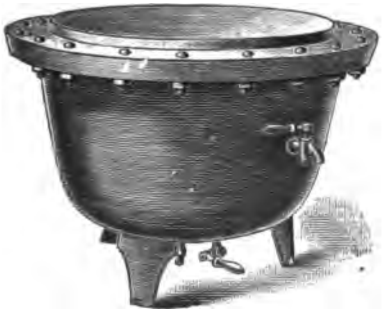
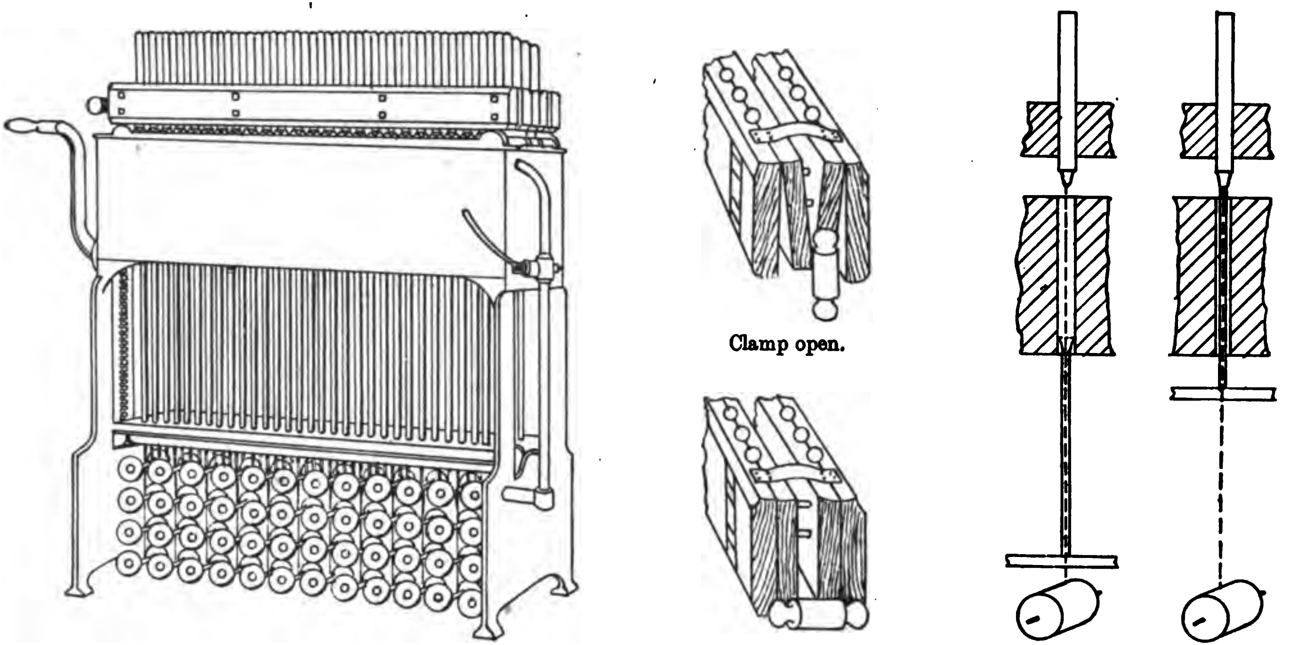


FIG. 98.—Melting pan.

The melting tubs are made of well-seasoned timber bound with iron hoops. The melted wax is run into these direct from the paraffin decolorising plant, and the stearine added in the solid state. If it is not convenient to transfer the wax in the liquid condition, the blocks are melted together with the stearine, open steam being used for the purpose. This usually contains a certain amount of lime salts, which will combine with the stearine. The mixture is therefore clarified by the addition of oxalic acid to precipitate any lime present. The impurities are run off along with the condensed steam. The clarified mixture is now run into a steam-jacketed enamelled iron or aluminium pan (fig. 98), in which any traces of water or other impurity will be readily detected and colouring matter added if the candles are to be tinted. Traces of water left in the candle will result in spluttering when it is burned, a defect which must be avoided.

Numerous tools are used by the candle maker, and these are illustrated by fig. 99. The first to be used is the "jack" or filling can, which may be provided with one or two spouts. The latter

ated by fig. 99. The first to be used is the "jack" or filling which may be provided with one or two spouts. The latter



Candle machine.

Clamp open.

Clamp closed.

Piston lowered.

Piston raised.

FIG. 100.—Candle-moulding machine.

admits of both troughs of the candle-moulding machines being filled at one time. Fig. 100 illustrates a complete moulding machine, the clamp mechanism, and the operation of the piston in the mould.

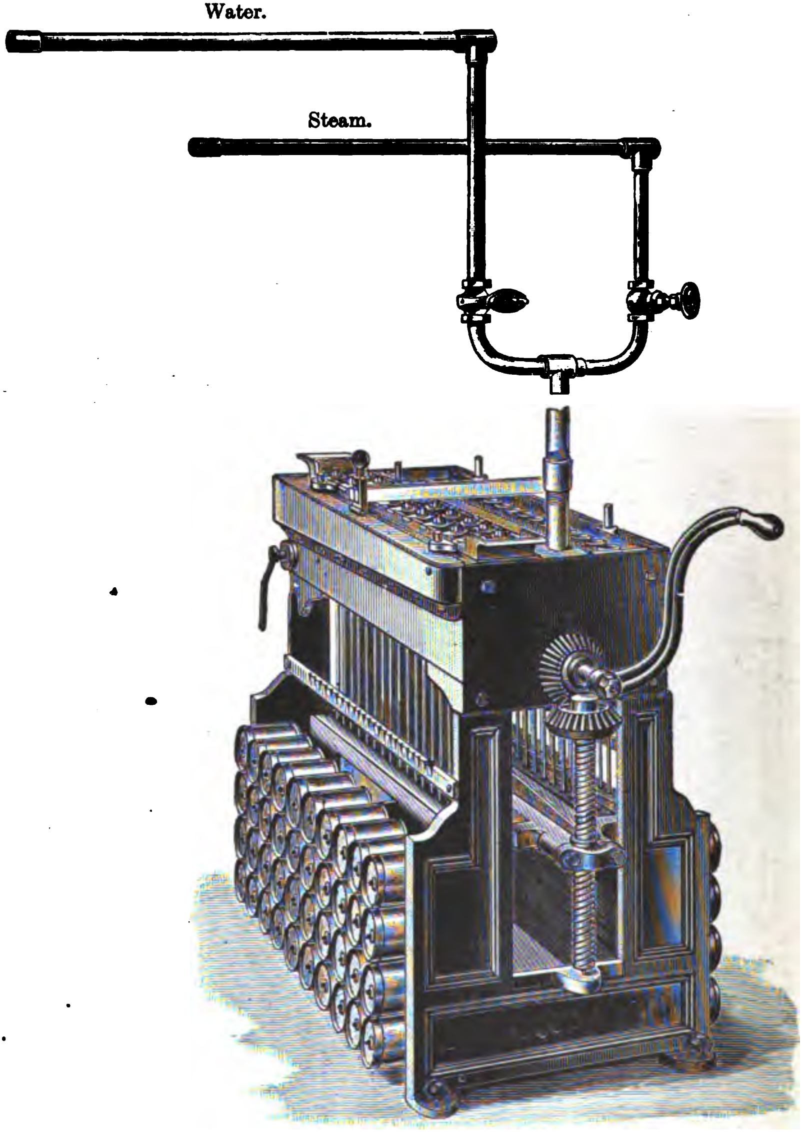


Fig. 101.—Candle machine, showing steam and water connections.

Figs. 97 and 101 show steam as well as water connected to the machines, but whether the former is required or not will depend on the climate in which the plant is to be operated. Hard wax candles containing sufficient stearine to ensure high bending-point may be manufactured satisfactorily in the tropics with cooling water as high as 90° F. Under such conditions no steam connection is necessary, but in temperate countries, where the cooling water is low in temperature, the moulds must be heated to a point between that of the water and the melting-point of the wax before being filled. A little

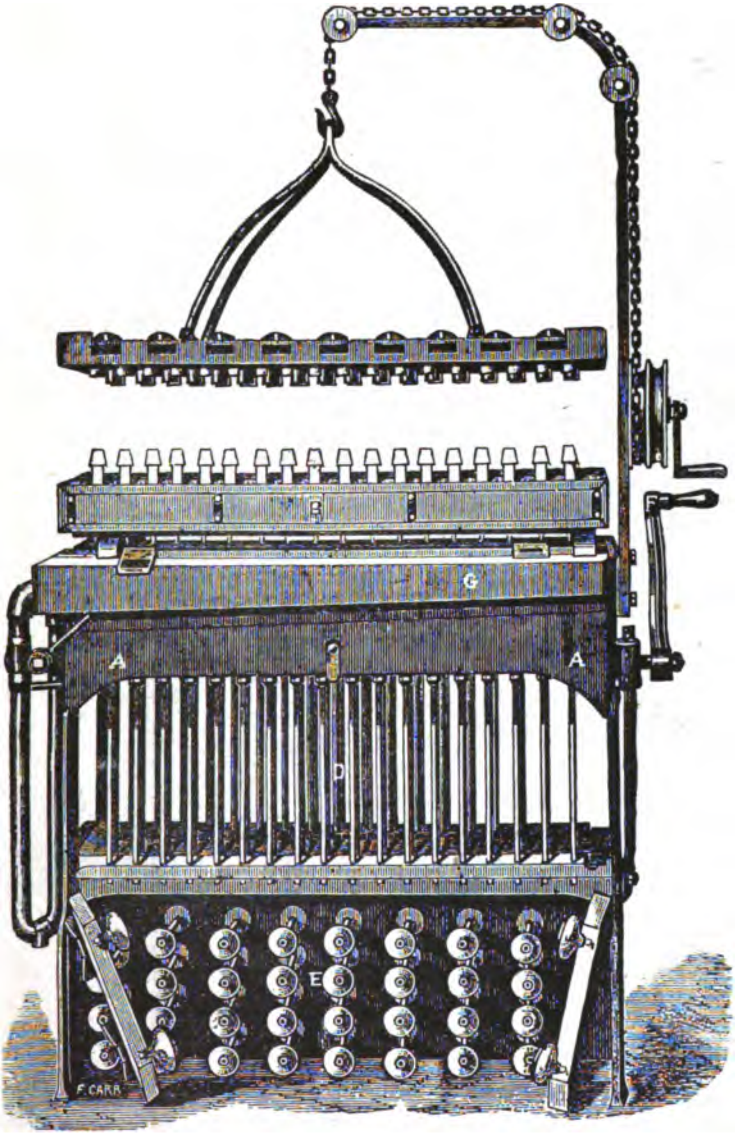


Fig. 102.—Candle-moulding machine (self-fitting).

practice will reveal the working temperature which yields best results. The out-turn from candle machines depends on the melting-point of the mixture and the temperature of the cooling water.

The machines must be filled from the jack, rapidly and steadily, without splash or spillage, and sufficient melted wax left in the troughs to allow the moulds to fill up as their contents contract on cooling. As soon as the machine is full, the cooling water is circulated until a proper degree of solidification is reached, and this also will be learned from experience of the material to be moulded. The surplus wax, now in a hard state, lying in the troughs, is removed with a "spud," care being taken to see that the material is sufficiently hard to retain the wick in a central position in the butt of the candles. The clamps

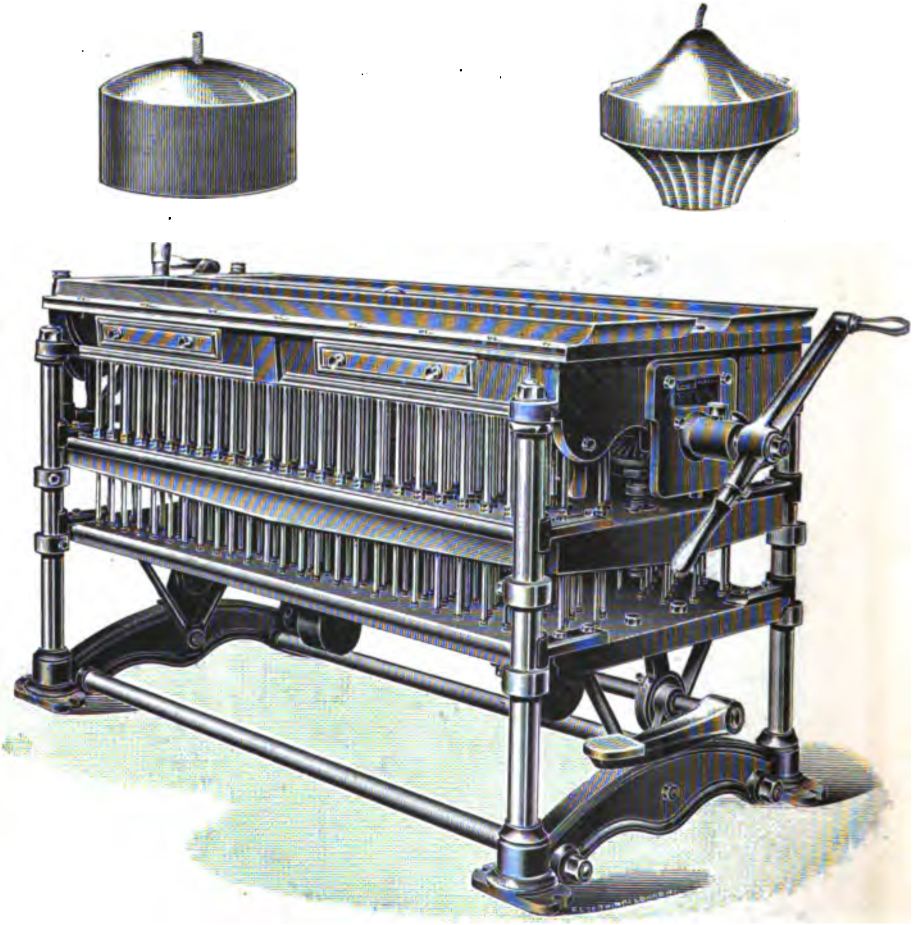


Fig. 103.—Night-light moulding machine.

are now placed in position, and the lifting gear operated by applying to it a steady and vigorous pressure which raises the candles from the moulds into the open clamps. The pistons are now at their highest position, and a catch applied to the pinion-wheel prevents their descent until the batch of candles has been shut into the clamps. This having been done, the catch is removed, the pistons lowered, and the wicks straightened in the moulds, when the machine is ready for the succeeding charge of melted wax. Filling and cooling are repeated, but this time the wicks must be cut before the surplus wax is removed, the candles transferred from the clamps to a tray, and the clamp returned to the machine before the batch is "lifted."

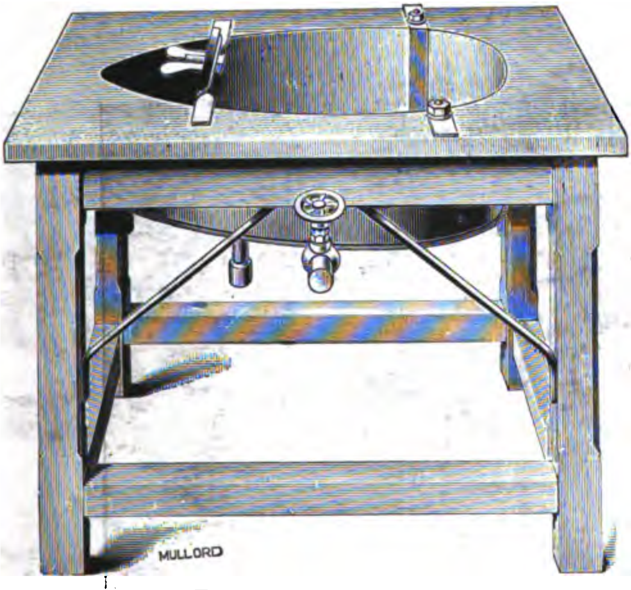


Fig. 104.—Draw-wick pan.

Considerable care is necessary in operating candle machines to prevent damage to moulds and piston tips.

Machines for making candles with "self-fitting" ends are fitted with a special removable plate for moulding the ends or butts, as shown by fig. 102.

The manufacture of "night lights" is somewhat different, the wick being threaded in and attached to an "anchor" by hand.

Fig. 103 illustrates a machine for making the "lights" and piercing them to receive the wick. Night lights are usually encased in cardboard, which retains the melted wax when the wick is lit. There are several types of night-light wicks, but the single cotton wick anchored to piece of tin is quite satisfactory. The prepared wick is coated with wax of a much higher melting-point than that of which the light is to be made, and this is done by drawing it through a pan (fig. 104), containing the wax coating. A reeling arrangement is necessary for drawing the wick evenly through the pan.

Candles are made in almost innumerable shapes and sizes to suit the markets in which they are to be sold. Fig. 105 shows a number of "fancy" shaped domestic candles.

Picture "transfers" are used for decorating candles, sometimes with most pleasing results, and when this is done, after the picture is transferred to the candle,—a somewhat difficult task—it is allowed to dry, after which the candle is dipped in melted hard wax. This gives the completed candle a very smooth appearance.

Wrapping of candles in papers, placing them in cardboard boxes or telescoping cartoons entails a great amount of labour, as also does labelling, etc.

The boxing of the packets, cardboard boxes or cartoons, also accounts for much labour, and so far this operation has not been performed by machinery.

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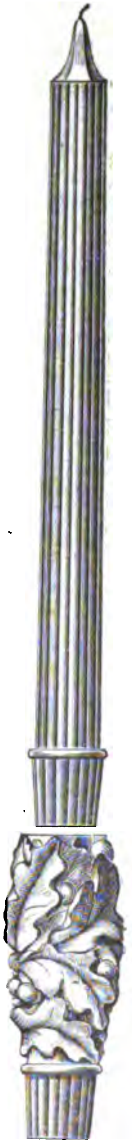

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CHAPTER VII.

CHEMICAL TREATMENTS.

THESE may be divided into three sections—those for (1) the light volatile fractions; (2) for kerosenes and intermediate oils; and (3) for lubricating oil base, heavy oil and paraffin, paraffin wax and high viscosity lubricating oils.

The vessels employed, called "washers," are cylindrical in form, erected either in an upright or in a horizontal position. For benzine the agitation is produced by the operation of mechanical stirrers; for kerosene and all the other oils air may be used as the agitating medium. To reduce evaporation to the minimum, benzine washers must be closed and a vapour outlet connected to a gasometer or absorption apparatus. For all the other oils it is unnecessary that the vessels should be gas-tight.

There are two reasons for treating oil with chemicals—(1) to improve the colour, and (2) to render it pleasant to the sense of smell. The distillates from some crude oils are so pale in colour and satisfactory in smell that they meet market requirements without treatment other than distillation. Some require only filtration through a decolorising medium, such as bauxite or fuller's earth, to render them of the desired degree of colour, and in this case an arrangement similar to that used for decolorising wax is used.

The chemicals mostly employed for improving the colour are sulphuric acid (concentrated oil of vitriol) and caustic soda solution, whatever fraction is to be treated. The amount used varies with the colour of the original oil; generally speaking, the darker the oil the greater the percentage of acid required. The oil, thoroughly freed from water, is filled into the washer and the necessary percentage of sulphuric acid (previously determined by experiment) added. Agitation is carried on for a given period, the heavier oils taking longer than the lighter. The contents are then allowed to settle until the tar subsides to the bottom. The latter is run off and conveyed to a special lead-lined tank, where it is washed free from acid and then, in a melted state, burned as fuel, as already mentioned in Chap. II.

The acid tar being separated, the oil is transferred to another washer, where any free acid it still contains is neutralised by an agitation with a weak solution of caustic or carbonate of soda. This part of the operation is sometimes troublesome with the higher viscosity oils, as they are inclined to emulsify, a condition difficult to remedy. After neutralisation and final washing with hot water the oil is transferred to shallow tanks of large area, where it is settled by steam heat. These shallow tanks or "bleachers" are arranged in buildings having a glass roof to admit as much light as possible; some oils, at this stage, being considerably "bleached" by the action of the sunlight. Lubricating oil washers are steam-jacketed and fitted with heating coils for the regulation of the temperature of the oil during treatment. For deodorising both petrol and kerosene fractions, litharge dissolved in caustic soda (sodium plumbite) is used. This treatment removes a certain amount of sulphurous compounds. It is a somewhat messy operation, as the sulphur combines with the lead salt and is precipitated as sulphide, forming a dark brown sludge which must be recovered. Permanganate of potassium is a suitable deodorising agent for some petrols and kerosenes, and if properly used gives good results.

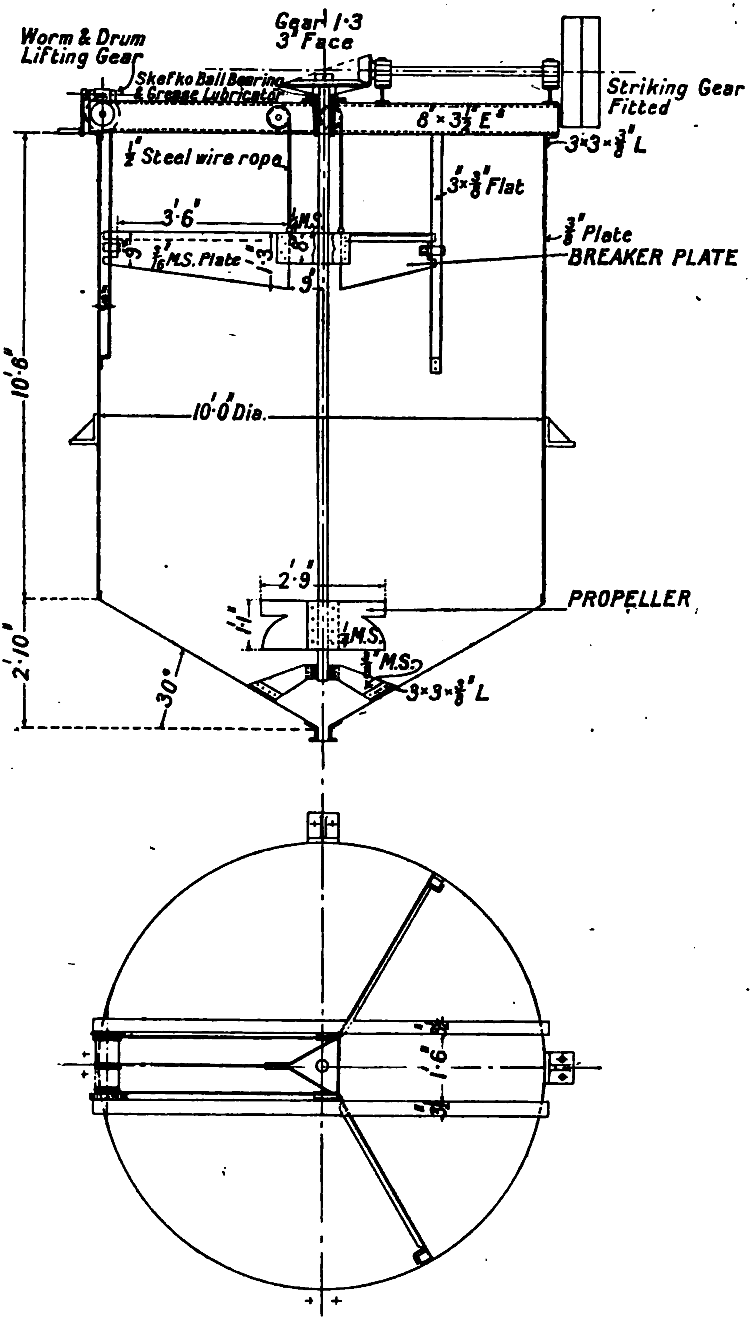
In treating lubricating oils, fuming sulphuric acid is sometimes necessary, and is generally followed by the usual C.O.V. treatment.

Washers for petrol and kerosene may be of almost any capacity, provided that the means of agitation is ample. For the heavy lubricating oils, the author has had no experience of a larger capacity than 100 barrels, and is inclined to think this a convenient size for working oils likely to emulsify. It is recommended that a separate vessel be employed for each chemical used for treating the lighter as well as the heavy oils.

In Chap. IX. constructional details of a vertical washer will be found.

The vortex system has been adapted to the washing of oils by Mr Robert B. Gray (Messrs Christie & Gray), and is very effective. It is claimed for it that the maximum of agitation is produced in the washer with the minimum expenditure in power. Fig. 106 illustrates this system of washing.

For the treatment of benzine a continuous washing plant has been suggested (English Patent, 29,833/1912), and is illustrated diagrammatically by fig. 107. The action of sulphuric acid, etc., appears to take place just as satisfactorily as when the chemicals and oil are lashed together by agitation. Referring to the illustration, it will be seen that the benzine to be treated is pumped into one or other of the vessels, A A, alternately, one discharging through the cylinders, B B B, containing the chemicals, while the other is being

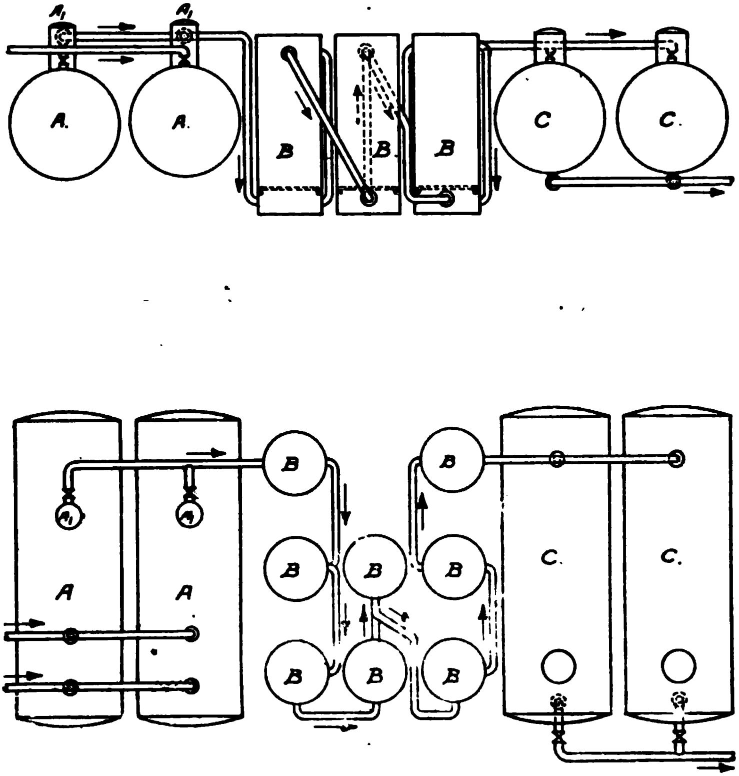


Full capacity, 5600 gallons. Working capacity, 5000 gallons.

Fig. 106.—Gray's patent typhoon agitator.

filled with the raw material. Water is pumped into A, gradually displacing the benzine at a regular rate, thus forcing it through the chemicals and finally into the receivers, C. The whole is under pressure, so that there will only be a minimum possibility of loss by evaporation during the treatment.

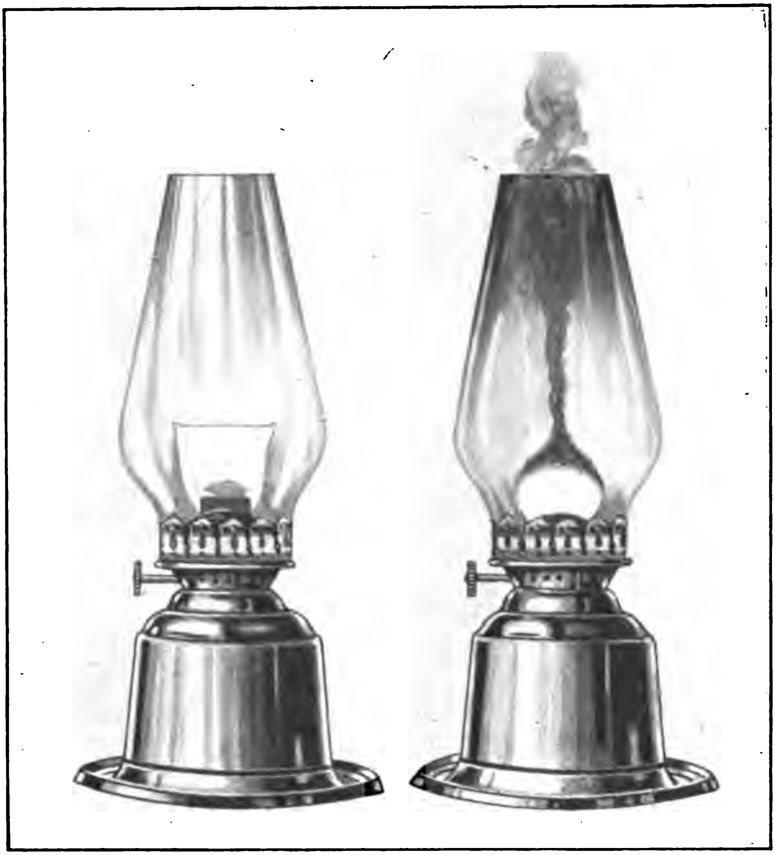
An outstanding development in the treatment of kerosene is the process for refining petroleum with liquefied sulphur dioxide, patented and worked successfully, on the large scale, by Dr Edeleanu, the eminent Roumanian chemist (British Patent, 11140/1908).



A A = Vessels containing untreated benzine which is displaced by water; $A_1 A_1$ = Dome provided with gauge glass to show when benzine is displaced; B = Vessels containing chemicals; C = Receivers for treated benzine.

FIG. 107.—Elevation and plan of continuous benzine washing plant.

The object of the process is to remove from kerosene those constituents which emit smoke when the oil is burned in an ordinary lamp. The process is a physical and not a chemical one, liquid sulphurous acid being a solvent for the smoke-producing constituents, leaving them undestroyed by chemical action, contrary to the effect when C.O.V. or fuming sulphuric acid is used for their removal. There seems to be some doubt whether the separation is effected quite perfectly, but from a refiner's point of view the result is satisfactory, as will be seen from reference to fig. 108, which shows lamps burning (1) untreated distillate of the "smoky" variety, and (2) the same distillate after the extraction of 15 per cent. "smoky" constituents with sulphur dioxide. The "extract" would not burn without emitting dense volumes of smoke. This process has also the advantage over the treatment with sulphuric acid in that it does not deteriorate the colour of the distillate nor produce the incrustation of lamp wicks so common with highly treated kerosenes. In the pamphlet entitled "The Edeleanu Process as applied to large



(After) (Before)

Fig. 108.—Lamps burning oil before and after SO_2 treatment.

Refineries," the principle and operation of the process is described as follows:—

"The operation of Dr Edeleanu's process depends on the peculiar property possessed by liquid sulphurous acid of dissolving the aromatic and other heavy hydrocarbons present in petroleum distillates. When such a distillate is shaken at low temperature with liquid sulphurous acid, the latter dissolves the above-named hydrocarbons, while leaving the paraffin hydrocarbons and naphthenes unaffected. Separation is simple, on account of the difference in specific gravity of the two liquids, in accordance with which they form two distinctly separated layers. The process of solution is as follows. A certain quantity of liquid sulphurous acid is dissolved in the petroleum distillate, the quantity depending on the temperature. Each temperature requires a certain degree of saturation; the higher the temperature at which the experiment is carried out, the more sulphurous acid is necessary for saturation. As soon as the point of saturation is reached, any further addition of sulphurous acid separates out with the aromatic and heavier hydrocarbons, whereby separation into two layers takes place. By continuing to treat the distillate with sulphurous acid, it becomes increasingly poor in aromatic hydrocarbons, until at last there is on the one hand a purified distillate, and on the other an extract which contains the aromatic hydrocarbons dissolved in the sulphurous acid. The purified distillate is almost free from aromatic hydrocarbons, and now contains much less dissolved sulphurous acid than at the beginning of the separation, owing to the gradual removal of those hydrocarbons, which are the particular solvents for the sulphurous acid.

"The richer a distillate is in aromatic and heavy hydrocarbons, the more sulphurous acid is required to remove the same. With rising temperature, the solubility of the paraffin hydrocarbons and naphthenes—which at lower temperatures are almost insoluble—increases rapidly; in contrast to the aromatic and heavy hydrocarbons, which are soluble in sulphurous acid at all temperatures. It follows from this that with a distillate rich in aromatic hydrocarbons, the most favourable conditions for treatment exist at a lower temperature; while, on the other hand, in the case of a distillate poor in aromatic hydrocarbons, the influence of temperature is not so noticeable. For instance, in the case of a Borneo distillate, which contains 46 per cent. of aromatic hydrocarbons, treatment with 66 per cent. liquid sulphurous acid at −7° C. produces no separation; but separation takes place to a considerable extent at −10°. In the case of a Mexican distillate, which only contains about 17 per cent. of aromatic hydrocarbons, treatment with an equal amount of sulphurous acid produces separation even at a temperature higher than +10° C. It will thus be observed that each individual distillate requires special conditions of treatment in order that the most favourable results may be obtained.

"The removal of the aromatic hydrocarbons from an illuminating oil distillate causes a lowering of the specific gravity and a raising of the burning capacity and illuminating power. The richer a distillate is in aromatic and heavy hydrocarbons, the more distinctly stands out the improvement brought about by treatment with sulphurous acid. The effect of the new process is clearly discerned when treating a Bustenari distillate. Whereas the chemical process hitherto employed is productive of a very inferior illuminating oil, which gives off a great deal of smoke even when the flame is turned down low, and which when burned in a 14''' cosmos burner develops only 7 candle-power, that illuminating oil refined by treatment with sulphurous acid is equal to the best American and Russian oils."

Then follows this table illustrating the action of the old and the new methods of refining :—

TABLE VIII.—Tests of Oils before and after SO_2 Treatment.

Origin of distillate.	Refined in the usual way with sulphuric acid and caustic soda.		Treated according to the Edeleanu process with sulphurous acid.	
	Specific gravity.	Candle power.	Specific gravity.	Candle power.
Roumanian oils—				
Bustenari	0·8195	7	0·8028	13
Campina	0·8148	10	0·7968	15
Moreni	0·8180	5·8	0·8048	11·8
Tintea	0·8190	8·4	0·8088	13·2
Galician oil—				
Tustanovice	0·8084	9·8	0·7970	13·7
German oil—				
Weitze	0·8190	10·5	0·8098	13·5
Russian oils—				
Grosny	0·8158	9·2	0·8072	14·7
Baku	0·8185	11·7	0·8145	13·8
Maikop	0·8158	6	0·7995	13
Burmah oil	0·8175	10·8	0·8007	16·5
Dutch-Indies oil—				
Borneo	0·8450	Burns in no lamp	0·8056	14
American oils—				
California	0·8190	6·5	0·8110	11·5
Peru	0·8167	10·0	0·8068	13·6
Mexico	0·8025	11·8	0·7926	14·4
Scotch shale oil	0·7950	9	0·7810	16·2

Regarding the utilisation of by-products and the effect of the process on distillates containing sulphur, the pamphlet continues :—

"The extract obtained in addition to illuminating oil contains, as mentioned, aromatic hydrocarbons and hydrocarbons rich in carbon. On account of their low-burning capacity they cannot be employed as burning oils; nevertheless, by dividing the extract into two products by redistillation, the lighter become turpentine substitutes or, mixed with light benzine, motor-spirit of high efficiency, and the heavier a motor oil for Diesel motors. The extract is, therefore, as regards the possibility of turning it to account, at least equally as valuable as the distillate.

"Further experiments have shown that the extract can be resolved by special means into the lower homologues of the aromatic hydrocarbons. By pyrogenic distillation the hydrocarbons contained in the extract are split up; and, besides a gaseous part, a large percentage of tar is produced. The latter is distinguished from the tar derived from stone coal by its higher content of the lower members of the aromatic series of hydrocarbons.

"It is further to be noted that liquid sulphurous acid also possesses the property of dissolving out from crude distillates certain sulphur-containing constituents. For instance, by treating a Mexican distillate of 0·803 specific gravity and with a sulphur content of 0·6 per cent., an oil is obtained which carries a percentage of sulphur of only 0·08 per cent., and yet another of 0·79 specific gravity and with a sulphur content of 0·46 could be reduced to a percentage of sulphur of 0·04."

Fig. 109 is a copy of the diagram illustrating the operations of the plant, which has been designed to treat as much as 500 tons of distillate per day of twenty-four hours, and consists chiefly of five apparatuses: two coolers for cooling the sulphurous acid and distillate to the desired temperature; a mixing vessel in which these liquids are mixed together, and in which the separation of the refined oil from the extract takes place; and two evaporating vessels for recovering the sulphurous acid from the two separated fractions. The distillate must be filtered through salt to thoroughly remove any traces of moisture before it enters the cooler. By the introduction of efficient cold-exchanging apparatus, the cold liquids running off from the mixer preliminarily cool the oil and sulphurous acid entering the system. The final temperature is reached in the coolers through the action of a refrigerator. After the sulphurous acid is added to the oil in the mixer, in the proper quantity, it quickly settles to the bottom, taking with it the aromatic hydrocarbons, and has now a brown colour. The sulphurous acid, plus the extract, is then pumped through the cold-exchanging apparatus into the evaporating vessel, fitted with steam-heated coils, and the bulk of the sulphurous acid driven off through a condenser and returned to the reservoir, from whence it

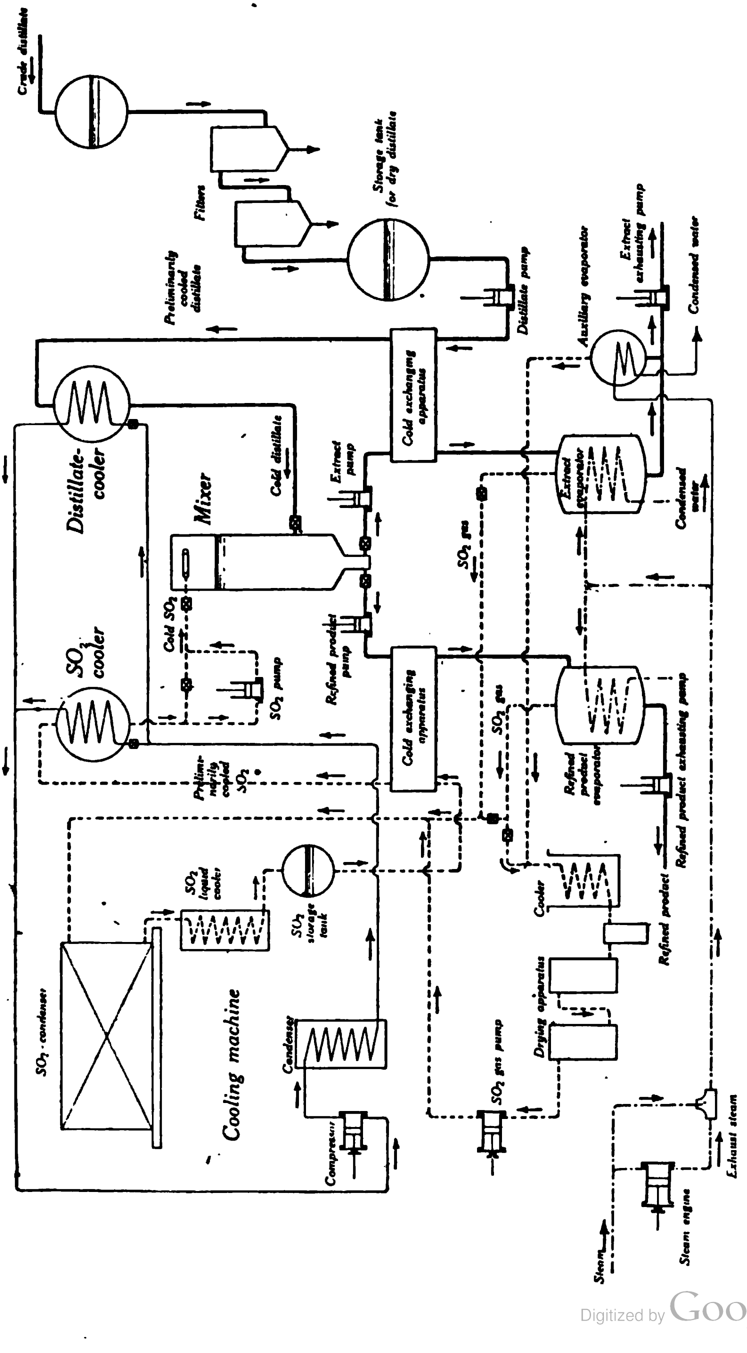


FIG. 109.—Scheme of a refining plant on the Edeleanu process.

enters on another cycle through the system. An auxiliary evaporator is employed to remove the final traces of sulphurous acid which

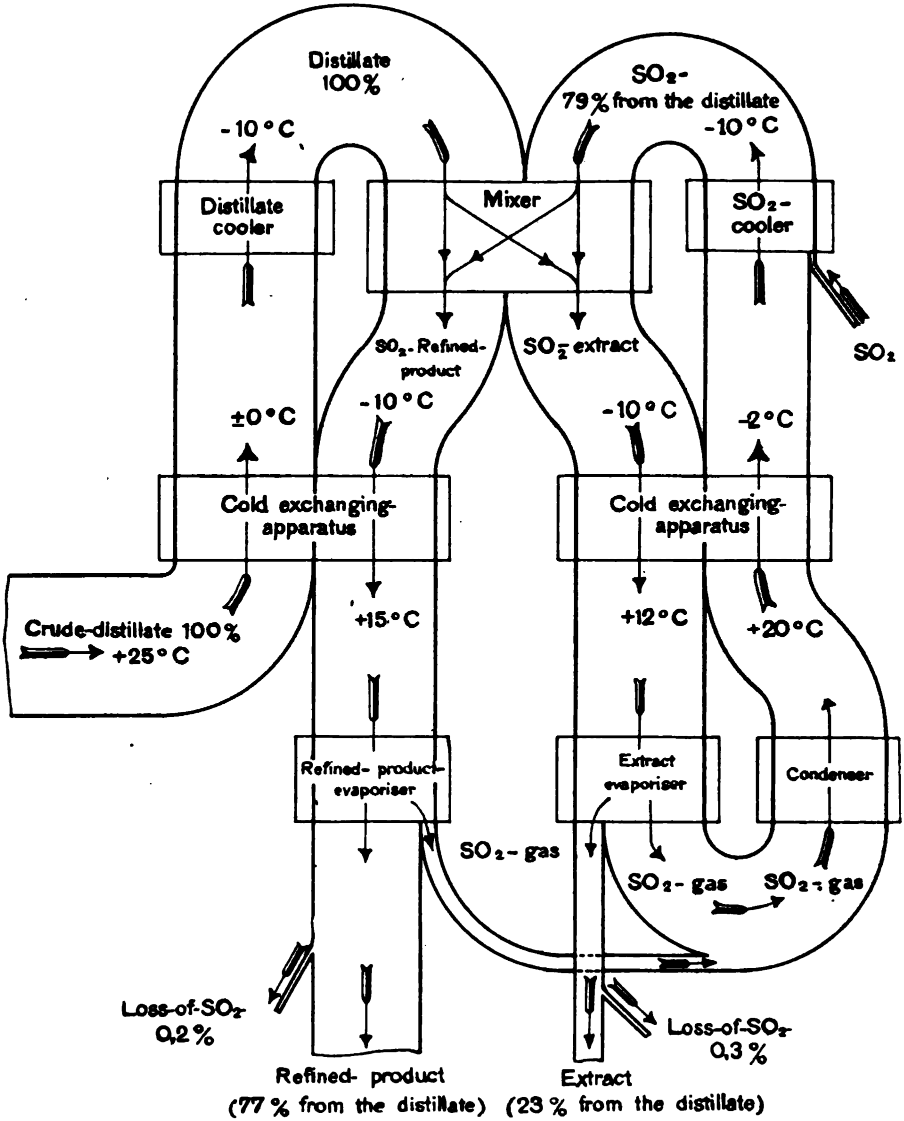


FIG. 110.—Scheme of the Edeleanu process (Roumanian distillate).

are difficult to remove from the extract, and this is done with the aid of a gas pump.

The refined oil passes through similar operations in the vessels arranged for their treatment, and is finally washed with an alkaline solution, and sometimes with a small quantity of sulphuric acid, if the colour is required as light as possible. The whole cycle of operations takes about sixty-five minutes. Care has to be exercised to prevent any moisture entering the system, for, while liquid sulphurous acid, in itself, is inactive towards metals, when water is added to it, it becomes a very powerful corrosive.

Fig. 110 illustrates the operation of the process on a Roumanian distillate.

Following are the statements of working results abstracted from the pamphlet above referred to.[1]

"The working results of a plant with an average daily capacity of treating about 65 tons of distillate per twenty-four hours, as were reached in the plant at Ploesti, are set out in the following table :—

"Working Results of a Plant with a Daily Capacity of 65 Tons.

Specific gravity of the crude distillate to be treated	0·820
Average time of operation	1 hr. 8 min.
Quantity of distillate treated at each operation	3115 kg.
Temperature in the mixer	—10° C.
Specific gravity of treated product	0·8028
,, ,, extract	0·8691
Sulphurous acid left in the treated product	0·16 per cent.
,, ,, ,, extract	0·36 ,,
Figures of consumption—	
Consumption of fresh steam per operation	1200 kg.
,, ,, ,, ,, 1000 kg. distillate	385 ,,
,, ,, exhaust steam per operation	648 ,,
,, ,, ,, ,, 1000 kg. distillate	208 ,,
Cooling water used per operation	17·1 cbm.
,, ,, ,, 1000 kg. distillate	5·5 ,,
Loss of sulphurous acid per operation	17·5 kg.
,, ,, ,, 1000 kg. (distillate)	5·6 ,,

"From the above figures, the cost of treating a Roumanian Bustenari distillate can be calculated as follows :—

"Cost of Treatment in a Plant of 65 Tons Daily Capacity.

Cost of plant—	
Machines and apparatuses	Frs. 130,000
Buildings, construction, freight, duty, and other expenses	,, 100,000
Total	Frs. 230,000

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[1] "The Edeleanu Process as applied to Large Refineries."

Amortisation and interest—

Amortisation = 10 per cent. of frs.	130,000	Frs. 13,000
,, = 5 ,, ,,	100,000	,, 5,000
Interest on the invested capital = 5 per cent. ,,	230,000	,, 11,500
Total		Frs. 29,500

In 325 working days, at 65 tons per day, there will be 21,125 tons treated, so that the amortisation and interest amount to frs. 1396 per ton, or per 100 kg. . . . . Frs. 0·140

Maintenance, labour, etc.—

Repairs, working materials	Frs. 6000
Wages and salaries	,, 8000
Insurance, 1½ per cent.	,, 3450
Lighting and heating	,, 2400
Drying salt, unforeseen expenses	,, 3000
Total	Frs. 22,850

The cost of maintenance and labour, therefore, amount to frs. 1·082 per ton, or per 100 kg. . . Frs. 0·108

Cost of materials—

There are required for each 1000 kg. of distillate:

385 kg. fresh steam	at frs. 3·50 per ton	Frs. 1·35
208 ,, exhaust steam	,, 1·50 ,,	,, 0·31
5·5 cbm. cooling water	,, 0·04 per cbm.	,, 0·22
5·6 kg. sulphurous acid	,, 18·00 ,, 100 kg.	,, 1·01

Cost of materials per 1000 kg. . . . . Frs. 2·89

,, ,, ,, 100 ,, ... Frs. 0·289

The total costs of treatment, therefore, amount to . Frs. 0·537 or practically frs. 0·54 per 100 kg. of distillate or 3·9 shillings per ton. It must also be taken into account that frs. 18·00 per 100 kg. of sulphurous acid is a very high figure, and refers to imported sulphurous acid. The cost of this material is very much lower when sulphurous acid is used which is produced in the country itself, or actually in the refinery.

“Cost of Treatment in a Plant of 500 Tons Daily Capacity.

Cost of plant—	
Machines and apparatuses	Frs. 1,100,000
Buildings, construction, etc.	,, 500,000
Total	Frs. 1,600,000

Amortisation and interest—		
Amortisation = 10 per cent. of frs.	1,100,000	Frs. 110,000
,, = 5 ,, ,,	500,000	,, 25,000
Interest on the invested capital = 5 per cent. ,,	1,600,000	,, 80,000
Total		Frs. 215,000

In 325 working days, at 500 tons per day, there will be produced 162,500 tons, so that the amortisation and interest amount to frs. 1323 per ton, or per 100 kg.	Frs. 0·132

Maintenance, labour, etc.—		
Repairs, working materials	Frs. 40,000	
Wages and salaries	,, 30,000	
Insurance, 1½ per cent.	,, 24,000	
Lighting and heating	,, 10,000	
Drying salt, unforeseen expenses	,, 20,000	
Total	Frs. 124,000	
Brought forward		Frs. 0·132
The cost of maintenance and labour, therefore, amount to frs. 0·763 per ton, or per 100 kg.		,, 0·076

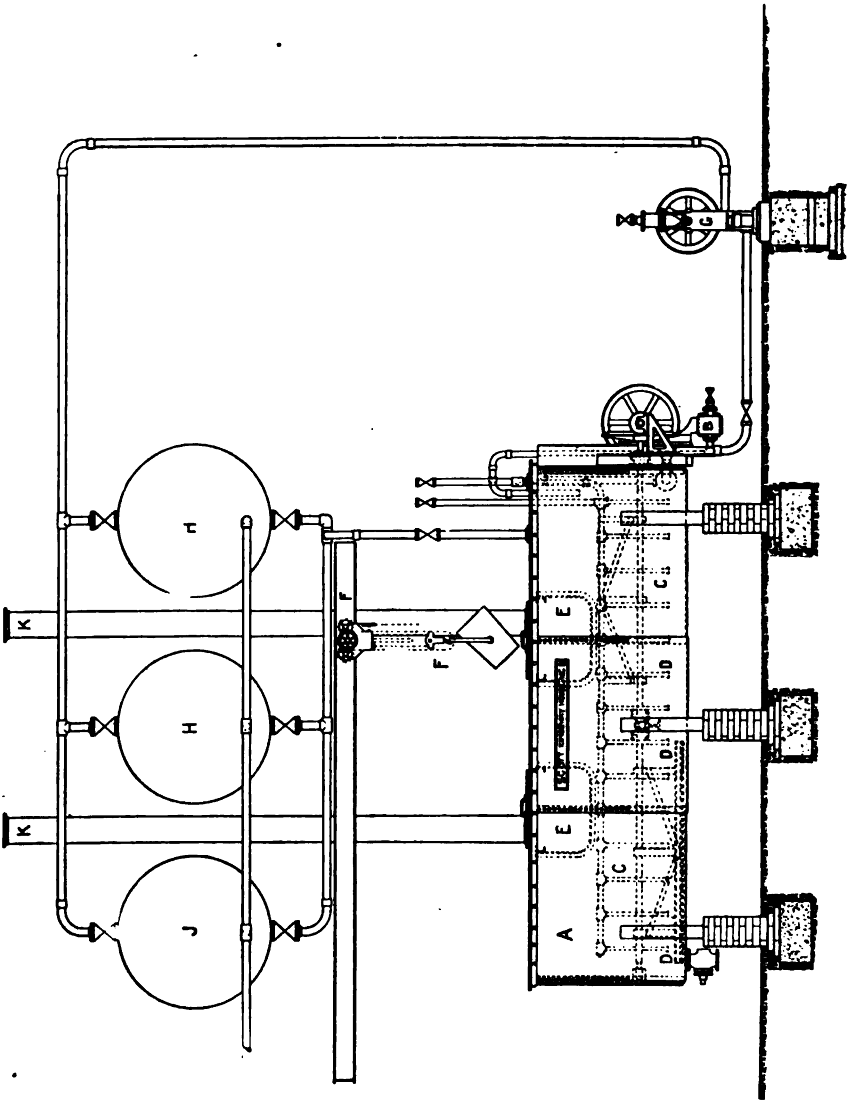
Cost of materials—

There are required for each 1000 kg. of distillate :

385 kg. fresh steam	at frs. 3·50 per ton	Frs. 1·35	
208 ,, exhaust steam	,, 1·50 ,,	,, 0·31	
5·5 cbm. cooling water	,, 0·04 per cbm.	,, 0·22	
5·6 kg. sulphurous acid	,, 10·00 ,, 100 kg.	,, 0·56	
Cost of materials per 1000 kg.		Frs. 2·44	
,, ,, ,, 100 ,,			Frs. 0·244

The total costs of treatment, therefore, amount to . Frs. 0·452 or roughly, frs. 0·45 per 100 kg. of distillate or 3·26 shillings per ton.

In this case, the cost of sulphurous acid is taken at frs. 10·000 per 100 kg., sulphurous acid produced on the spot being reckoned. The cost of production is amply estimated throughout.



A = Causticiser; B = Driving engine; C = Agitators; D D = Steam jets; E = Lime cages; F = Lime and alkali hoist and traveller with tipping skep; G = Liquor pump; H H = Clear liquor tanks; J = Lime washing water used for succeeding charge; K K = Vents for waste steamy vapour.

FIG. 111.—Soda recovery plant.

"The above tables are based on figures actually obtained from the existing plants. It is, however, to be anticipated, that with further perfection of the working of the process, the costs will be reduced even more."

The manufacture of sulphuric acid and caustic soda are frequently included in the modern refinery.[1]

When raw materials are available, some refiners adopt the process, employed in other branches of chemical industry, for the preparation of the caustic soda liquor at the strength actually consumed instead of purchasing solid caustic. The raw materials consist of alkali (sodium carbonate) and lime. The process is briefly one of dissolving the alkali, boiling and gradually adding the lime.

The plant which is almost universally employed for this liquid caustic is that made by Messrs George Scott & Co. (Ltd.), and one example of which, suitable for 18 Twad. liquor, is illustrated diagrammatically herewith (fig. 111).

For liquor stronger than 18 Twad. an evaporator is required in addition.

The profit saved in normal times is about £3 per ton of caustic, which would otherwise be bought. A 10-ton plant is stated to be capable of repaying cost of instalment in first year's work.

---

[1] Information regarding these processes will be found in *Sulphuric Acid and Alkali*, George Lunge, Ph.D., and in other works of reference.

CHAPTER VIII.

DISTRIBUTION OF PRODUCTS.

LIQUID products may be despatched from the refinery to the consumer either direct in barrels, drums or tins, or in small bulk in railway tank wagons or by tank motor lorry. The refinery may not be situated at a convenient distance from the market to be supplied

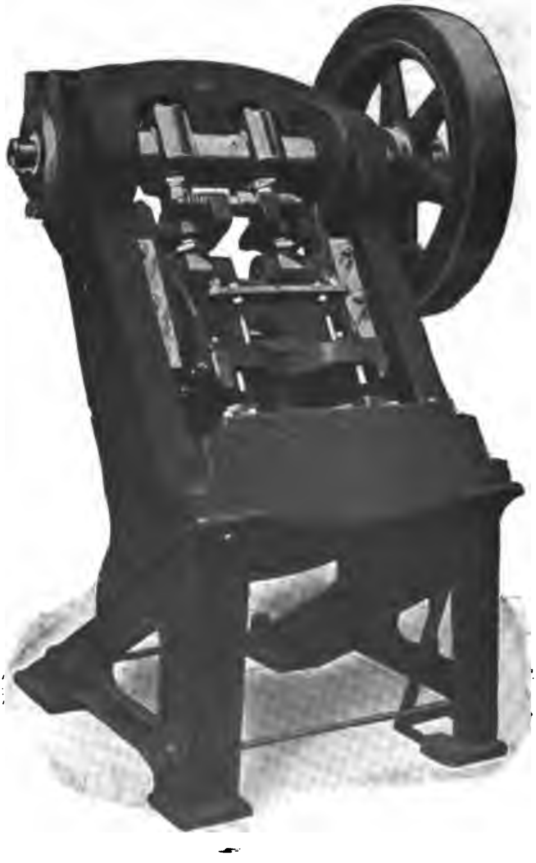


FIG. 112.—Body trimming press.

to permit of this being done economically, and in that case the liquid products will be despatched in tank steamers to bulk installations, erected at favourable points, and where distribution in smaller quantities, as above, will be undertaken. Circumstances will therefore decide whether the machinery for making up packages will be erected at the refinery or at bulk installations.

Petrol is usually packed in tins to contain 2 imperial gallons, drums (4 imperial gallons), and steel barrels (40–65 imperial gallons).

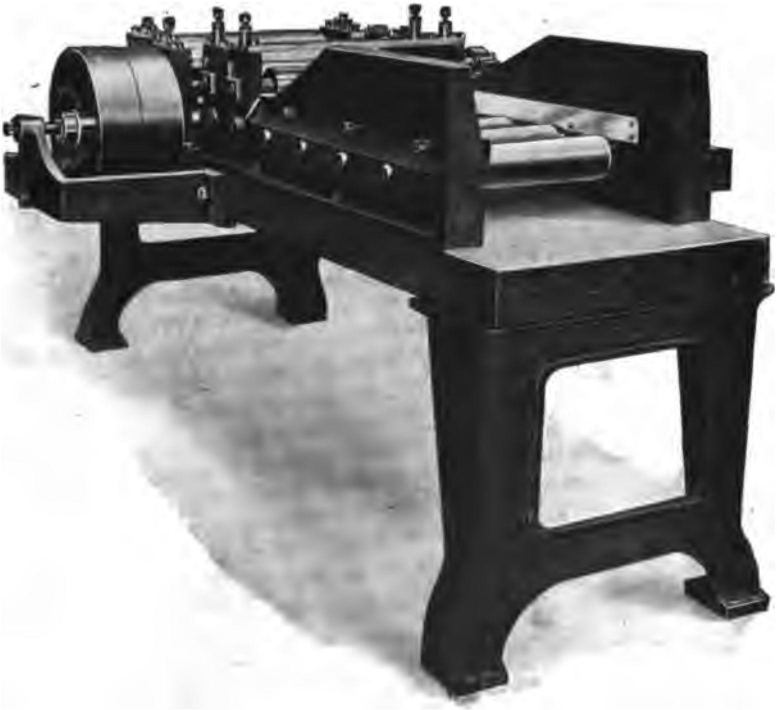


FIG. 113.—Roll-hemming press.

The steel plates used for the manufacture of petrol tins and drums are much thicker than those for kerosene tins, and each package is fitted with a screw plug which ensures a petrol-tight joint. The storage and distribution of petrol and kerosene in bulk and packages are in many countries controlled by Government, or are subject to railway companies' regulations, and the rules laid down with regard to the form of package and the materials of which it is to be constructed should first be ascertained and must be observed.

Petrol is sometimes distributed in 4-gallon tins, but these, owing to the lightness of the tin sheet of which they are made, are packed in pairs in a wooden case.

Kerosene is packed in tins to contain 4 imperial gallons, or in wooden or steel barrels. The tins may be delivered in cases containing two tins, if circumstances necessitate the additional expense. Lubricating and intermediate oils are generally distributed to large consumers in 40-gallon (imperial) wooden barrels, and to the small consumer in tins or drums of varying capacity. Drums containing 4 and 5 imperial gallons are the usual capacity.

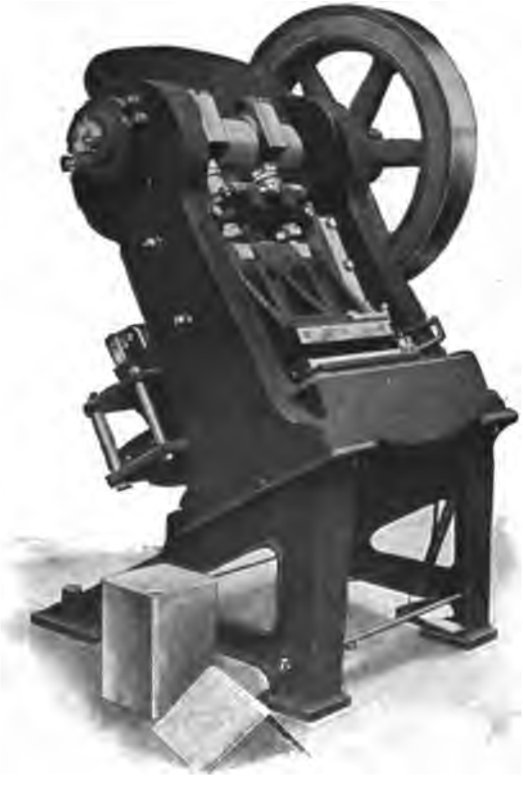


FIG. 114.—Panelling and bending press.

The manufacture and filling of the petrol tin, the kerosene tin, and the drum are all on the same lines, the class of tin sheet used being the chief difference. A description of the manufacture of the "hemmed edge" kerosene tin, therefore, will suffice for all. Three sheets are required to form one tin, and these are made in standard sizes. Two sheets, the "body blanks," form four sides, and one the top and bottom. The body blanks are cut to the exact size in the trimming press (fig. 112), the cut or "trimmed" sheet then passes through the roll-hemming machine (fig. 113), which, by turning over the edges, gives greater rigidity to the completed tin.

The sheets now pass to the panelling and bending press (fig. 114), which stamps the panels, hooks or folds the edges (for the vertical joint), and automatically bends the body at right angles at one operation. In the illustration the bent side and the complete tin are to be seen. The sides now pass on in pairs to the horning or grooving press (fig. 115), which securely joins or locks the vertical seams of the body, one seam at a time. A machine for joining the two seams in one operation is also made. The body is now complete.

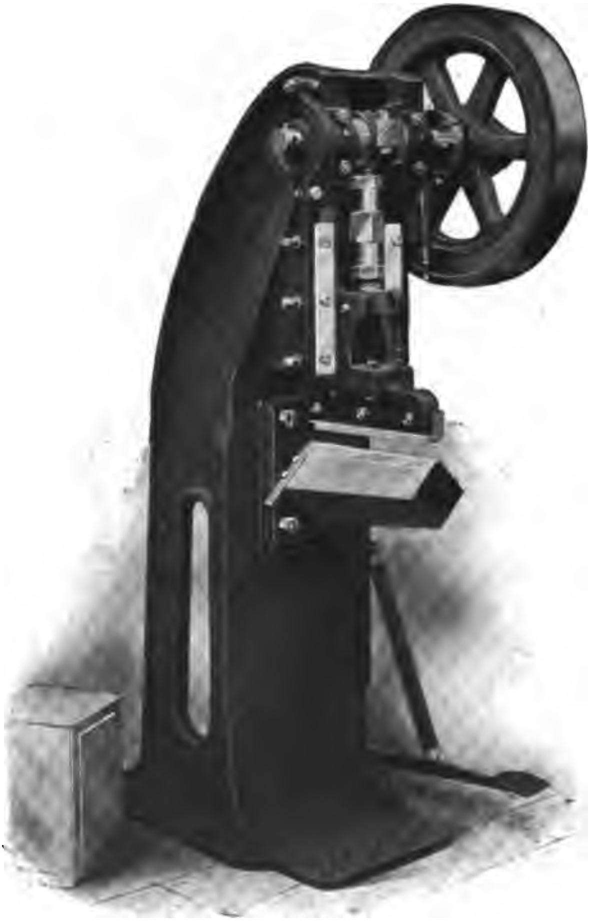


FIG. 115.—Grooving and horning press.

The third sheet, forming the top and bottom, is cut and embossed by a power press (fig. 116), in which the die for forming the bottoms or tops may be fitted alternately. There are innumerable designs for tin tops. Fig. 117 shows several well-known designs.

Google

The top and bottom are now attached to the body in the squeezing machine (fig. 118). The tin, now completely formed, enters the seam-rolling machine specially designed for economy in solder, which it accomplishes by securely pressing the seam between the top and bottom and the body (fig. 119). The screw caps, high or low shapes, are made in stamping, screwing, and crimping machines, the latter operation being that of attaching the bottom. Fig. 120 shows these two sizes of caps. The plain flat cap is made by stamping it from the sheet in a small press at one blow. The tin handle (fig. 121) requires several operations, stamping, beading, and rolling, to form it.

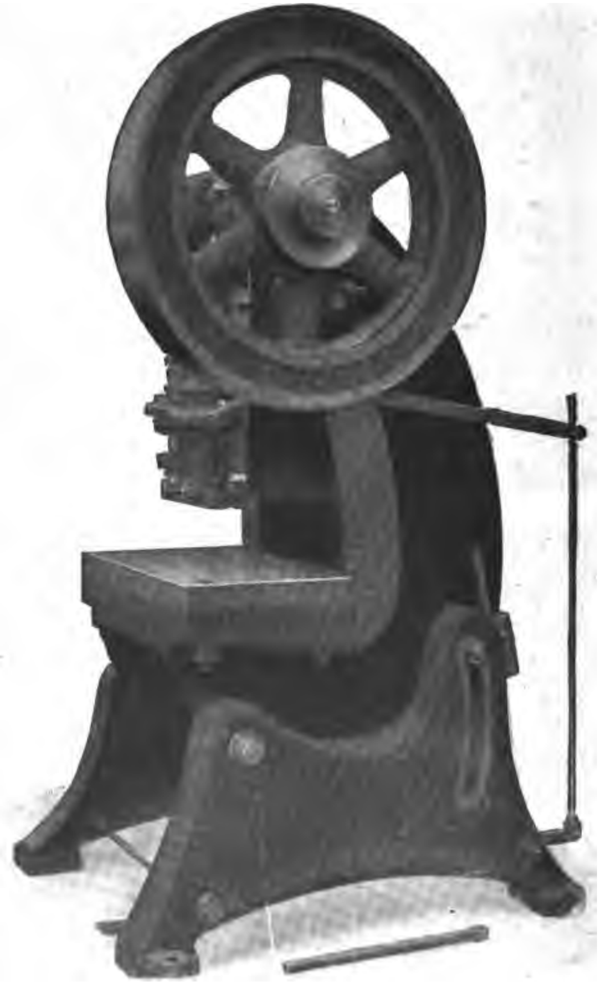


FIG. 116.—Top and bottom cutting and embossing press.

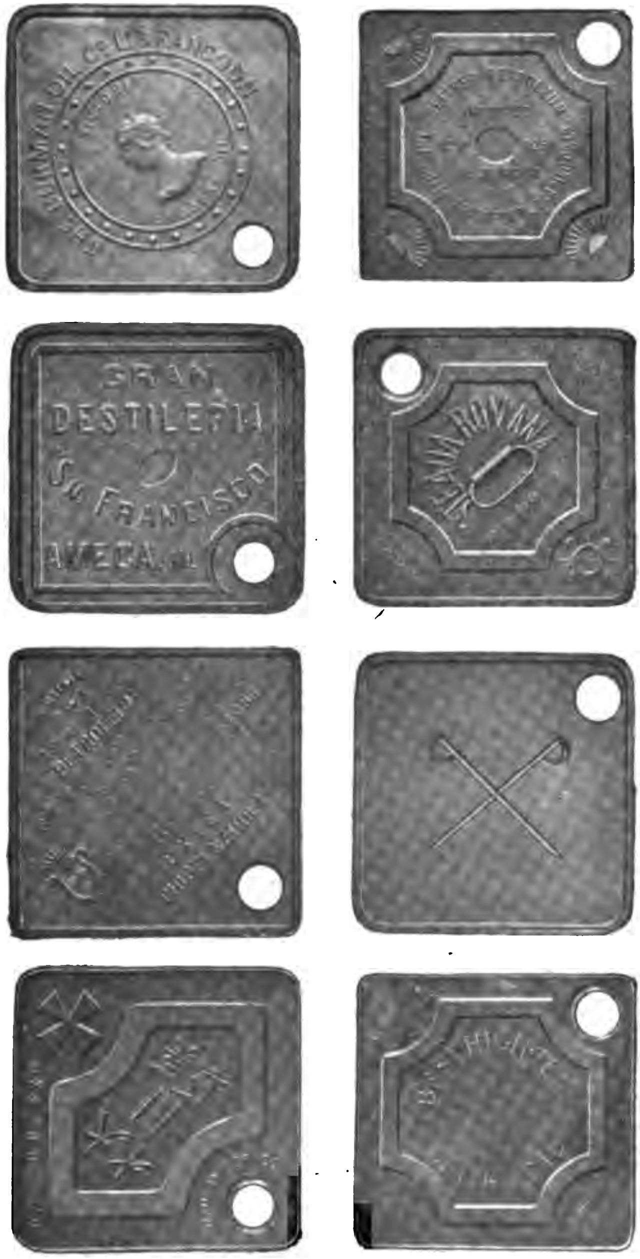


FIG. 117.—Tops of cans.

The automatic wire-handle-forming machine is very ingenious, and that illustrated by fig. 122 completes 85 oval wire handles per minute. The tins are now soldered and the top and bottom seams are dipped in the dipping stove (fig. 123), which may be heated by either solid, liquid, or gaseous fuel, while the side seams are soldered by hand. The Averay Jones soldering machine (Eng. Pat., Asiatic Petroleum Co., Ltd., and H. Averay Jones, No. 21,830/1914) is an invention wonderfully successful in saving both labour and solder.

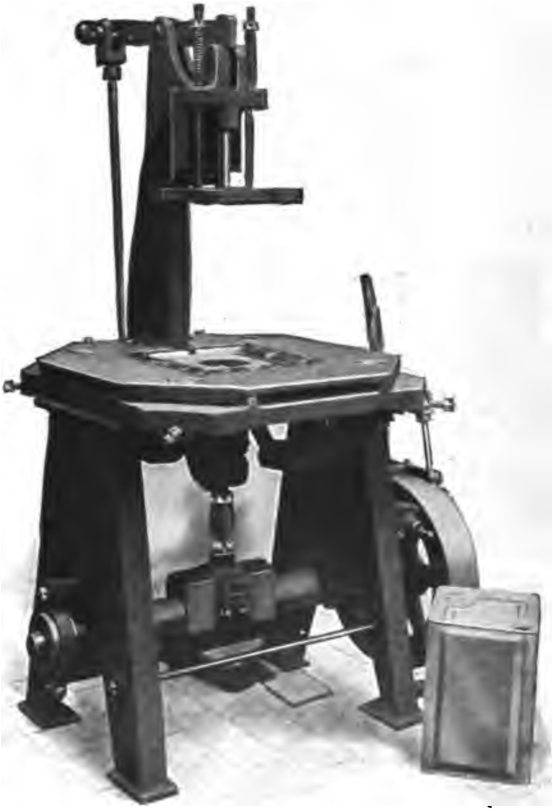


Fig. 118.—Squeezing machine.

The last operation in this department is to fill and close the tin with the cap. This is done on a filling machine, of which there are a number of designs. Fig. 124 illustrates a useful and convenient apparatus designed by Mr H. Simmons. Figs. 125 and 126 are fillers, by the same designer, for 2-gallon and 4-gallon petrol tins. The former is arranged to admit of two grades of spirit being filled at the same time. The screw, or flat cap, is soldered on by hand to close the tin.

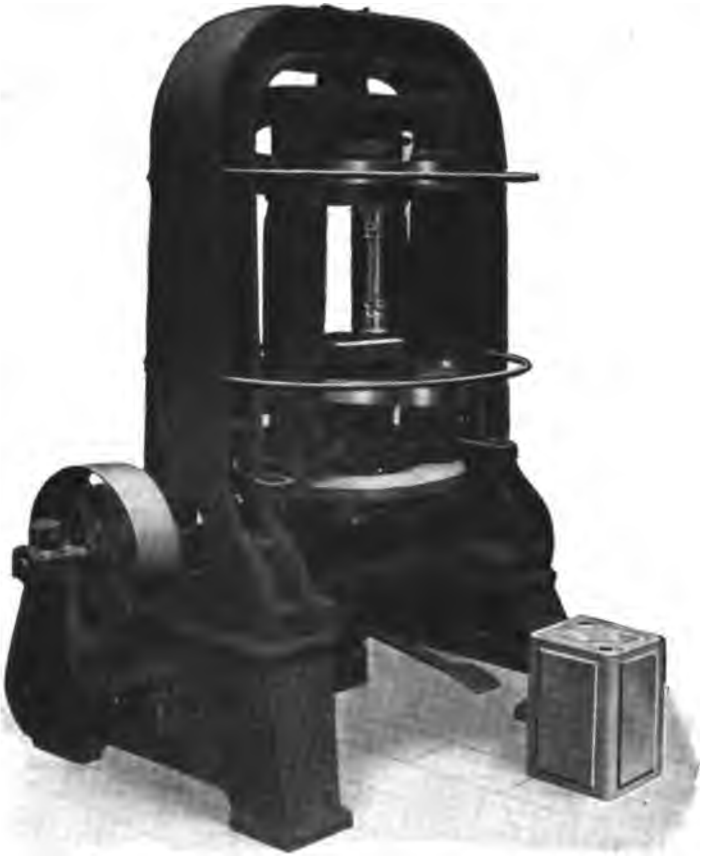


FIG. 119.—Rolling machine.

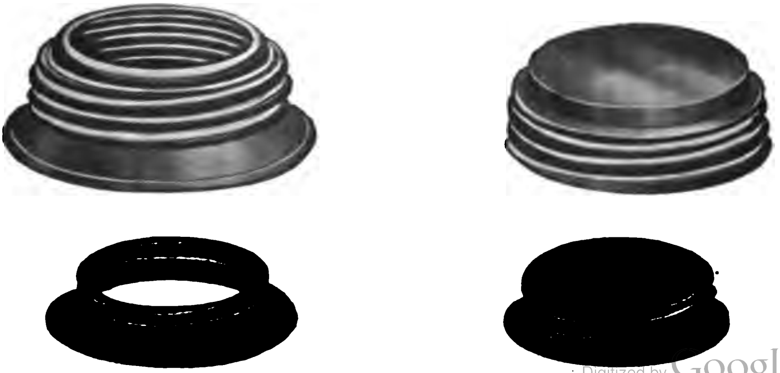


FIG. 120.—Screw caps.

The machine fig. 124 is for filling ten 4-English or 5-American gallon tins at a time. A central compensating tube passing through the whole line of measures is adjustable by the lever attached to the indicator plate, between the end of first measure and operating wheels.

FIG 121.—Tin handle.

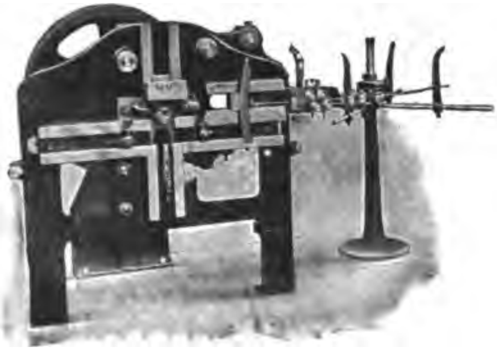


FIG. 122.—Wire-handle machine.

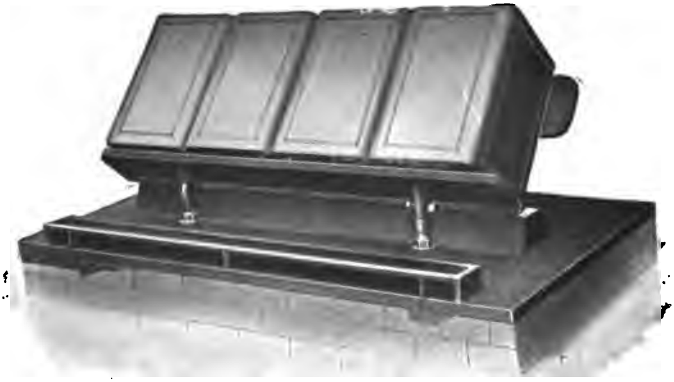


FIG. 123.—Dipping stove.

The quantity delivered can thus be varied, enabling liquids of different gravities, or varying temperatures, to be filled into the tins at a fixed weight.

tins

The measures and all moving parts are correctly tooled to gauge. The heavy gun-metal outlet cocks are of special vertical pattern, with spring packed glands. Sliding sleeves fitted to the tail pipes of the cocks automatically descend through the opening in the tins as the cocks are turned on by a half-turn of the side hand-wheel.

The square table (built of steel) revolves on light wheels, fitted with ball bearings, round a fixed pivot attached by radial bars to an angle iron ring forming a wheel race.

In working, empty tins are placed on one side of the table, while those on the front are being filled, caps are being soldered on the full tins on the opposite side, and at the same time those at the back are being removed. The table is turned round one side for each operation, and is brought to rest in the correct position for filling by the locking gear, as shown.

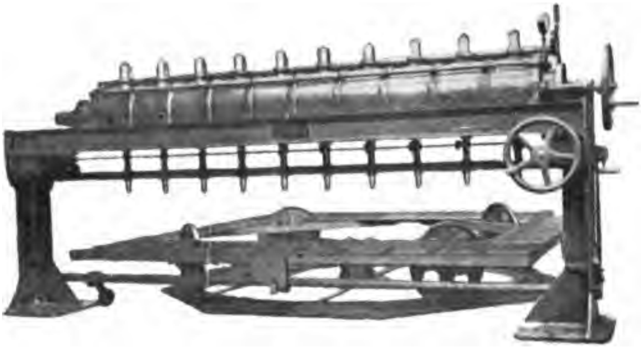


Fig. 124.—Filling machine (four gallons).

The operation of filling and emptying the measures occupies from thirty to forty seconds.

The double- and plain-seam tins are also largely employed for the distribution of kerosene, etc., and the machinery for making them differs slightly from that described above.

Some makers test all petrol and kerosene tins and drums with air pressure to ensure that no " leakers " reach the filling machines, and in the modern tin factory all forms of conveyors are employed to reduce labour to a minimum.

Petrol tins are returnable for refilling, and must be cleaned. For this operation there are several designs of machine, that of Mr H. Simmons being very efficient for removing moisture, grit, and other foreign matter (fig. 127).

It consists of a strong galvanised tank, with a false bottom. A piston hand-pump of large capacity is mounted on one end of the tank, and a filter pipe at the other supports a galvanised iron trough.

A longitudinal pipe connected with the pump carries a number of jets or sprays through which the petrol is pumped in a multitude of powerful jets, washing the entire internal surface of the inverted tins. The petrol as it leaves the tins is caught in the trough, and passing through the filter returns to the tank and is used over and over again. A gland cock is fitted to the tank for drawing off.

Two-gallon petrol tins and 4-gallon drums are painted. They are dipped in a trough containing the paint mixture, allowed to drain, and then "stoved" to render the coating as hard as possible. Tins which have been in circulation, and consequently tarnished, are washed free of the old paint in a soda solution, dried, and repainted before refilling.

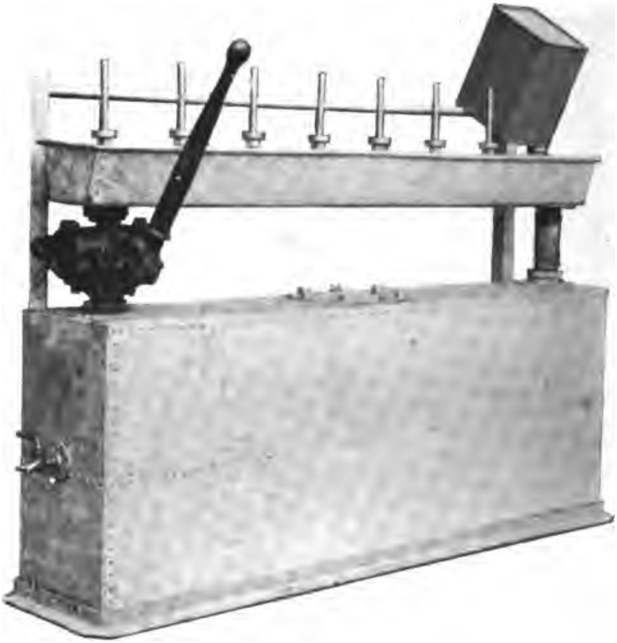


FIG. 127.—Petrol tin washing machine.

The welded steel drum or barrel of varying capacity is a reliable package for the transport of inflammable liquids. The Steel Barrel Co., Ltd., Uxbridge, Middlesex, are makers of these and all forms of welded tanks for petrol storage. Their system of petrol storage and measurement, for garages and works where petrol is used, is exceedingly simple, which in itself is a great recommendation. The spirit is stored in an underground welded tank which will not leak, and can therefore be set in concrete. It is galvanised, after being otherwise completed, which absolutely prevents rust and depreciation, and keeps the spirit clean. This storage tank is filled either from a tank wagon or from steel barrels in which the spirit is supplied. When delivered by tank wagon, the flexible hose, always carried by such vehicles, is screwed on to the tank wagon outlet cock at one end and the other end to the filling pipe of the storage tank. This ensures the latter being filled under seal. The filling pipe contains a filter to keep all dirt out of the storage tank. The quantity received is checked by dipping it with a carefully graduated rod. If the spirit is delivered in steel barrels, apparatus is provided to pump it, under seal, from these into the storage tank through the same filling pipe, and in this case an additional filter is provided to prevent any dirt getting into the pump.

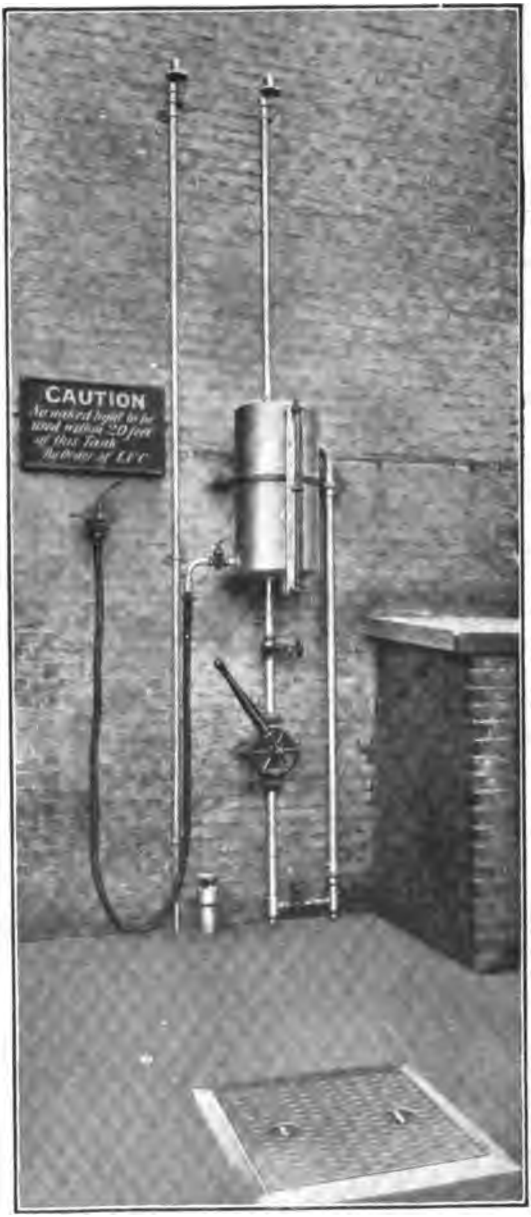


Fig. 128.—Underground welded tank. Petrol storage.

A semi-rotary pump is used to raise the spirit from the storage tank into the measuring tank, from which it is delivered through a flexible hose to the vehicles, the quantity being read off on a gauge.

The accuracy of the measuring tank is guaranteed by the fact that all measuring tanks are stamped after careful test by an officer of the Board of Trade. All the connections are under lock and key.

There is no leakage or waste of spirit, which is handled rapidly and cleanly, and, as it can be bought much more cheaply in bulk than in small packages, considerable economy is effected. Fire premiums are less costly for storing and distributing in this way.

Fig. 128 illustrates the arrangement described. The welded tank is situated beneath the manhole door in the foreground. Fig. 129 is a 1000-gallon welded petrol tank suitable for a road motor wagon.

The wooden barrel is largely employed for distribution. It contains 40 imperial gallons, and is used for kerosene and all grades of oils up to the heaviest lubricants. These barrels are made in the United States and shipped to all parts of the world, where they are bought second-hand by local oil producers. If they are intact they will only require steaming, drying, and gluing, and the hoops driven up by hand perhaps, to make them fit for further service; but if the staves have come apart, or should they be very dirty and are taken apart for scraping, the barrel must be re-formed and the hoops driven on tight. Fig. 130 illustrates an apparatus in which staves are "set up" to form a barrel. The hoops are fitted on at this stage, and the ends put in. Fig. 131 is a machine, worked by hydraulic power, for permanently

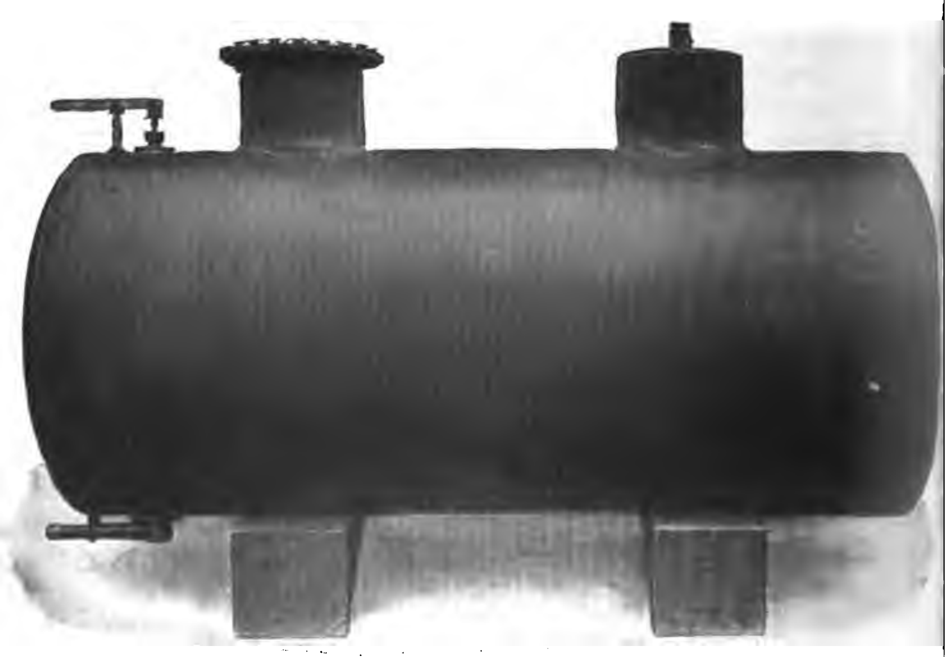


FIG. 129.—Welded petrol tank for road motor tank wagon. Capacity, 1000 gallons.

driving on hoops, which is easy to work and very efficient for the purpose. It is provided with a large number of arms which have a parallel motion, so that the faces of the drivers which come in contact with the hoop are always in a horizontal position. As each arm is independent of the others, the drivers will catch the hoop no matter how much the barrel may be out of the round. The pressure can be adjusted to drive the hoops perfectly tight, and without danger of breakage.

This machine not only effects a great saving in hoops and an enormous economy in labour, but it hoops the barrels far more tightly and evenly than can be done by hand.

In dealing with second-hand casks it may be necessary to make a large proportion of new hoops, and for that purpose labour-saving machines are obtainable.

The manufacture of new casks entails the setting up of extensive machinery requiring expert advice. Oil casks are made of American oak, and it has not been possible to obtain timber in other countries satisfactorily to compete with it.

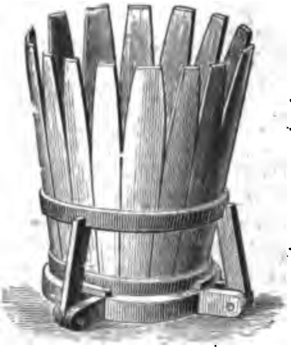


FIG. 130.—Setting-up apparatus for barrels.

The cleaning of dirty barrels is accomplished by inverting the barrel over a steam jet which enters the bung hole, the washings dropping into a trough which also forms the support for the barrels. An air current, blown over a steam coil (enclosed) or other means of heating, is driven gently through the barrels, carrying with it any moisture adhering to the inside. When the barrels are "glued," the glue solution is introduced while hot, the barrel turned over in all directions, and the excess solution drained out while still hot. After the glue has "set," the barrel is again dried. The painting and marking of the ends (trade name, weights, and shipping marks) take up a considerable amount of floor space, but this may be economised by storing the full barrels, ready for delivery, on racks, three or four tiers high, to which they are raised by a barrel hoist. Filling may be direct from the tanks, the quantity being checked by deducting the tare from the gross weight, and this is the usual course with thin oils. When an exact quantity must be filled, measuring chambers are introduced between the tank and the barrel, and, if worked in pairs, filling will proceed without interruption. Fig. 132 is a design of measuring chamber adjustable to deliver either 39 or 49 gallons into the barrel. Automatic valve fillers are employed which prevent spillage. The

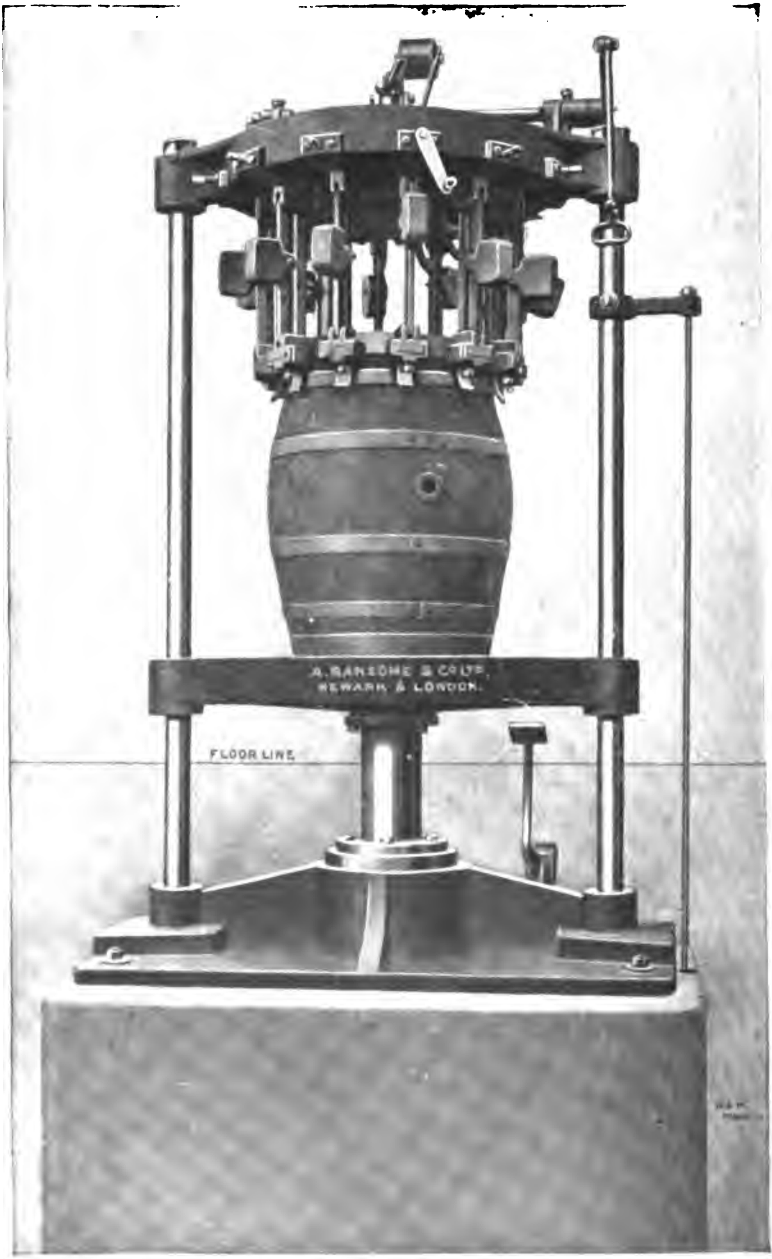


FIG. 131.—Hoop-driving machine.

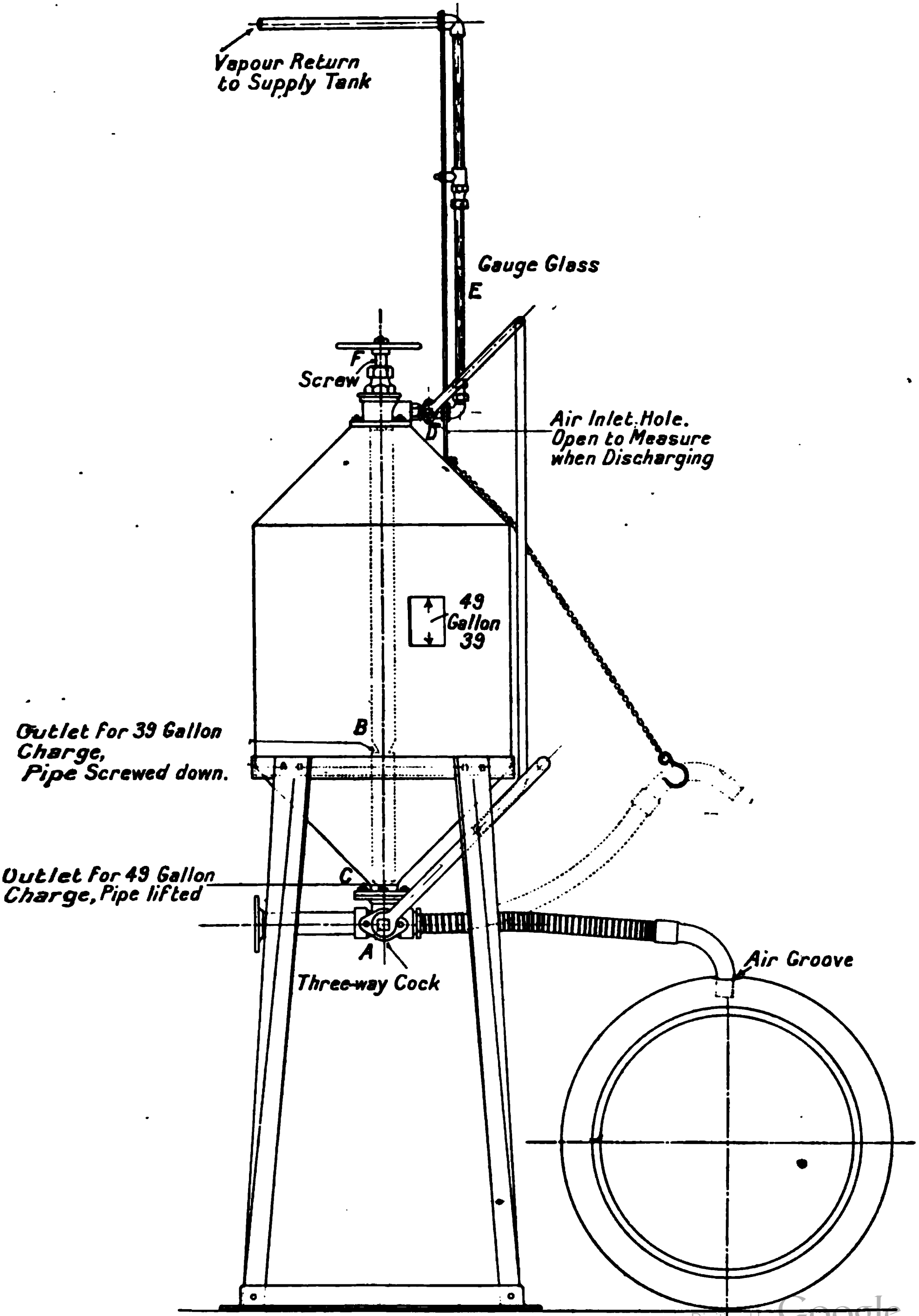

Vapour Return to Supply Tank
Gauge Glass
E
F
Screw
D
Air Inlet Hole. Open to Measure when Discharging
49 Gallon 39
B
Outlet for 39 Gallon Charge, Pipe Screwed down.
C
Outlet for 49 Gallon Charge, Pipe lifted
A
Three-way Cock
Air Groove

FIG. 132.—Adjustable barrel filler.

general arrangement of this department will depend on the contour of the site. Barrels may be loaded into rail wagons from the floor-level of the filling shed, or, if levels so necessitate, they may be loaded into the wagon by means of a barrel hoist, operated either by hand or mechanically. Heavy lubricating oil when despatched in small packages is filled by pumps which deliver the exact quantity required.

Many grades of lubricating oils may be delivered in bulk by tank steamer or wagons, but whether this is possible or not will depend, as already said, on the quantities to be sold and the situation of the refinery.

CHAPTER IX.

ENGINEERING SPECIFICATIONS.

SPECIFICATION OF STEEL TANK TO CONTAIN TWO MILLION GALLONS OF OIL.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one steel tank, 116 feet diameter by 30 feet 8 inches deep inside, all in accordance with this specification and with figs. 133, 134, and 135.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide the foundation under the tank, and will supply water for the first test of the tank bottom and the final test on completion. If the tank requires a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Plates.—The bottom plates will be $\frac{1}{4}''$ thick, with an annular outer ring $\frac{3}{8}''$ thick. The sides will be in six courses, of which the first will be $\frac{5}{8}''$ thick, the second $\frac{9}{16}''$, the third $\frac{7}{16}''$, the fourth $\frac{5}{16}''$, and the fifth and sixth $\frac{1}{4}''$.

Curbs.—The bottom curb will be of $5'' \times 5'' \times \frac{5}{8}''$ angle steel set to the required circle, machined on butts and covered with steel plate covers of section equal to the curb and not less than 2 feet long. The butting of angles must be a perfect fit; no wedging will be allowed.

The top curb will be of $3\frac{1}{2}'' \times 3\frac{1}{2}'' \times \frac{3}{8}''$ angle steel, set to the required circle. This curb to be machined on butts and jointed with steel plate covers of section equal to the curb and not less than 2 feet long.

Holes in Bottom.—On the bottom of tank there will be five openings for lowering, each fitted with a stiffening ring and covered with a plate fixed by studs.

Riveting.—The riveting will be as in Table IX. on following page.

In all plates and angle curbs the rivet holes must be drilled to template. The holes in crown sheets may be punched in multiple machine.

All the joints will be lap joints. The rivet holes in all bottom plates and in the horizontal flange of bottom curb are to be countersunk on upper surface.

The rivets will be hammered down inside of bottom. The $\frac{7}{8}''$ rivets will have countersunk holes and be hammered down outside, the other rivets being hammered down or " knobbled " on outside.

The rivet holes in plates and angles must coincide accurately when the work is put together, and the plates must be properly drawn together before riveting. No badly aligned work will be accepted and no drifting will be allowed.

TABLE IX.—PARTICULARS OF TANK RIVETING.

	Diameter of rivets.	Pitch.	No. of rows.	Distance between rows.	Lap.
	inches.	inches.		inches.	inches.
1st course sides—vertical .	⅞	3½	treble	1⅝	6⅛
2nd ,, ,, ,, .	⅞	3	double	1¾	4½
3rd ,, ,, ,, .	¾	2¾	,,	1½	3⅞
4th ,, ,, ,, .	⅝	2¼	,,	1¼	3¼
5th ,, ,, ,, .	⅝	2¼	,,	1¼	3¼
6th ,, ,, ,, .	⅝	2¼	,,	1¼	3¼
1st to 2nd—horizontal .	⅞	2½	single	..	2¾
2nd ,, 3rd ,, . .	¾	2¼	,,	..	2⅜
3rd ,, 4th ,, . .	¾	2¼	,,	..	2⅜
4th ,, 5th ,, . .	⅝	1¾	,,	..	2
5th ,, 6th ,, . .	⅝	1¾	,,	..	2
Bottom ¼-inch plates to ¼-inch	⅝	1¾	,,	..	2
,, ¼-inch ,, ,, ⅜-inch	⅝	1¾	,,	..	2
,, ⅜-inch ,, ,, ⅜-inch	¾	2¼	,,	..	2⅜
Bottom angle to bottom plates	¾	3¼	double	1½	..
Bottom angle to side plates .	⅞	4	,,	1½	..
Top angle to side plates .	⅝	2½	single	..	..
Crown sheets to top curb .	½	2	,,	..	..
,, ,, together . .	¼	1½	,,	..	1
,, ,, to centre plate .	½	1½	,,	..	1½

Caulking.—The edges of all plates and of the bottom angle curb are to be planed for caulking metal to metal. No cloth or jointing of any description will be allowed in any of the joints. The corners of plates are to be machine thinned at the three-ply joints; no thinning by heating and hammering will be allowed.

Crown Sheeting.—The sheeting of the crown will be No. 12 B.W.G., riveted together and to the top curb. The seams are to be jointed with prepared cloth dipped in red and white lead, and the crown when finished must be gas-tight.

The centre plate will be 6 feet diameter by $\frac{5}{16}$″ thick.

Framing.—The sheeting of the cover is to be supported on a framing of fourteen main rafters, fourteen intermediate rafters, one circle of main purlin bars, and seven circles of intermediate purlins.

The main rafters are attached at the centre to a framework of angles, flats, and plates forming a centre post.

The bottom members of the main rafters will be tied together by one circle of angle bars.

All the dimensions for the roof framework are detailed on fig. 134. Throughout this framework all rivet and bolt holes are to be drilled. Rivets are to be used wherever possible, and bolting will only be allowed on purlin bar ends and ends of main and intermediate rafters next centre.

Fittings.—The fittings to be supplied with the tank are shown on fig. 135, and include the following:—

One external ladder from ground-level to top of tank with intermediate stays to tank and 3½″ × ⅜″ flat piece at top fixed to the handrailing. At the top of the ladder there will be two special angle standards forming part of the handrailing. The ladder will be 15″ broad and of the sections shown on drawing.

A handrailing will extend round the circumference and will consist of two rows of 1″ bore wrought-iron tube, with forged wrought-iron standards. Twelve of the standards are to be tapped at top and to be fitted with an extension piece 18″ long with sharp points.

Two top mandoors 18″ × 15″ in opening with cast-iron frame and dished steel door complete with hinges, hasp, and padlock. The bearing surfaces are to be machined and scraped so as to give a tight metal-to-metal joint, and they must be perfectly gas-tight. For each door there will be provided two bars 2½″ × 2½″ × ¼″ angle steel, stretching from the outside of tank to the first row of purlins. These are to carry the weight of the door. One of the doors must be placed over the swivel pipe.

Two side mandoors each 24″ diameter in opening made of 6″ × 3″ channel steel frame and ¾″ dished steel cover plate. The channel frame is to be welded and machined on outside surface. The door is also to be machined so as to make a thoroughly tight job. All holes are to be drilled.

One set of swivel pipes 12″ diameter and of the dimensions shown on drawing. Each set is to be complete with pipes, wire rope, hood, and winch. The stool outside tank is to be of cast steel. As the winch has to sustain a considerable weight, the greatest care is to be given to its construction. The worm is to be of forged steel machined from the solid and the worm wheel of cast steel. The gland round the swivel pipe is to be packed with asbestos, which must be provided, and the joints made with asbestos millboard ⅛″ thick.

Four 6″ vacuum and pressure valves of approved type to be fitted on crown.

Four flanged dipping cocks 3″ diameter to be fitted to crown, of cast-iron gland pattern, and of full bore so as to offer no projection which might prevent the dipping rod passing freely.

Testing.—The bottom of the tank is to be tested with water before lowering, the depth of water being at least one foot. After completion the tank will be filled to the top with water and allowed to stand full for at least forty-eight hours. Under both conditions the work must be perfectly tight.

The water will be provided by the purchasers for one filling of the bottom and one filling of the tank, but any additional water required, owing to leakage or defects in the tank, must be at contractor's expense.

Painting.—Before delivery the whole of the material is to be oiled with pure boiled linseed oil. After all the surface has been properly scraped and cleaned and all scale removed, and before lowering, the bottom is to receive two coats of paint of approved quality.

The painting of exposed surfaces after completion will be undertaken by the purchasers.

GENERAL NOTES.

Quality of Material.—The material throughout must be of the best quality and most suitable for the purpose designed. The workmanship must be of the best class.

The tank is to be constructed of best selected mild steel capable of withstanding a tensile stress of 28 to 32 tons per square inch, with not less than 20 per cent. elongation in 8 inches.

The castings must be sound and free from defects of all kinds, and where machined must present a solid surface.

Preparation of Work.—To the utmost extent practicable contractors must complete work in their own premises, so that the amount of work to be carried out by them on the site may be reduced to a minimum.

Extent of Contract.—It is to be understood that this contract is to be complete in itself; that everything necessary for its completion, except such portions as are expressly excluded in the specification, are to be supplied and fitted by the contractor. All such furnishings and fittings as bolts, nuts, washers, grummets, jointing material of suitable nature, hangers, brackets, supports, etc., are to be supplied. If any detail shown on drawings is omitted in specification, or *vice versa*, or if any essential is omitted from both, it is to be understood that such omission is to be made good by the contractor and is to be included in his price.

Erection Marks on Tanks.

Various systems of marking for re-erection have been adopted where tanks have been partly erected and have to be shipped abroad.

The system shown on fig. 136 has proved quite satisfactory. In the case of a tank to be shipped and erected at a distance from place of manufacture the bottom plates, bottom angle curb, and first course of side plates should always be erected and marked before dismantling. The crown sheets, crown framing, and top course of sides should be erected complete and marked. The intermediate courses of sides need not be erected, but a section the full depth of tank should be laid out and marked with a broad painted band from top to bottom. It is a considerable help in picking out plates if each be marked with the course to which it belongs.

When a number of tanks are ordered, distinguishing colours should be used for painting as well as a distinguishing letter which should be marked on each piece belonging to the tank.

Tanks for Benzine and Petrol.

The foregoing specification may be used for tanks containing crude kerosene, or fuel oils. For tanks to hold benzine and petrol the specificatio should be more stringent.

The top curb should be inside the tank, as shown on the alternativ sketch on fig. 134. The crown sheeting should be riveted at closer pitch.

The roof should be absolutely gas-tight under a pressure of 6 inches water.

The top manhole should be made of steel plate with a bolted cover.

Vacuum and pressure relief valves may be fitted, or the crown connecte to an external vessel or regulator which will control the pressure on th surface of the liquid in the tank.

The riveting of side plates should be closer than for other tanks. Rive ¾″ diameter should be pitched at 2″ or 2⅛″ centres.

Great care should be taken in drilling the plates and in riveting at sit All the bottom plates and the heavier plates of the sides should be counte sunk and the rivets well hammered down. The plates should be plan

on edges for caulking inside and outside. It is preferable on account of caulking that no plate be less than 5⁄16″ in thickness.

All seams and joints where possible should be accessible. Although expensive, it may be advisable to set the tank on walls about 4 feet from ground-level so that the bottom may be examined.

Generally, too much care and attention cannot be given to the manufacture and erection of a tank to hold benzine or petrol.

No paint should be put on the plates until after erection and testing.

SPECIFICATION OF CRUDE OIL STILL 10 FEET DIAMETER BY 30 FEET LONG.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one still, 10 feet diameter by 30 feet long inside, all in accordance with this specification and fig. 52.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide the brickwork setting for the still, and will supply water for the test of the still. If the still requires a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Still.—The still will be 10 feet diameter by 30 feet long, and will be made up of ⅝″ and ½″ shell plates and ⅝″ end plates. Each end plate will be in one piece. These plates will be riveted together with ⅞″ rivets single at 2½″ pitch for circular seams, and double at 3″ pitch on horizontal seams, as shown in detail on fig. 52. The rivet holes will be drilled in position through two or more thicknesses. After riveting, all seams will be caulked oil-tight against a test pressure of 25 lbs. per square inch.

The end plates of still will be stayed to the shell by gusset plates and angles as shown.

In the still there will be fitted two horizontal flues of corrugated type, each in four lengths riveted together and to the end plates. These flues will be 3 feet diameter outside the corrugations and 2′ 8″ inside on the smallest diameter. The flue plates will be 7⁄16″ thick. Rivets for flues will be ¾″ × 2⅛″ pitch, and the flues will be welded longitudinally.

Mountings.—On the top of still there will be fitted one dome 3 feet diameter by 4 feet high, and one manhole frame 24 inches diameter with cover. On the front plate under the flues will be fitted one 15″ × 11″ M'Neil manhole and door.

The still will also be fitted with one 6″ diameter oil inlet pipe carried inside and to near bottom of still, and with one 6″ diameter oil outlet pipe carried across length of still with bell-mouth opening, with 6″ inlet and outlet valves and by-pass valve, as shown on fig. 52.

The still will be fitted with one 3″ diameter steam pipe carried to bottom of still and ending in a perforated tube lying along below flues. One 3″ steam valve to be fitted to this pipe in position shown.

One special plug valve with rod and guide to be fitted in the run-off pipe. One set oil-level gauges to be supplied and fitted, these to be of best class obtainable, of cast steel, and to have glasses 1″ outside diameter. Four spare glasses are required for still.

One 2½″ bore equalising pipe with 2½″ cock to be fitted near dome.

One 3″ deadweight safety valve of enclosed type and of approved design to blow off at 5 lbs. per square inch, with cast-steel stool to be fitted to the dome.

All the stools for these various fittings are to be of cast steel.

SPECIFICATION OF DISTILLATE PREHEATER, DEPHLEGMATOR, AND SUPPORTING FRAMING.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one distillate preheater, 6 feet 6 inches diameter by 23 feet long, one dephlegmator, 3 feet diameter by 8 feet long, and supporting framing, all in accordance with this specification and fig. 54.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide all foundations and will supply water for the test of the vessels. If the vessels require a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Distillate Preheater.—The preheater will be 6 feet 6 inches diameter ×23 feet long, and will be made up of $\frac{3}{8}''$ shell plates and $\frac{1}{2}''$ end plates. Each end plate will be in one piece. An opening is left in one end for insertion of the coils. This opening will be covered by a $\frac{5}{8}''$ plate riveted on after the coils are in position. The plates will all be riveted with $\frac{3}{4}''$ rivets $2\frac{1}{4}''$ pitch for both circumferential and longitudinal seams. The rivet holes will be drilled in position through two or more thicknesses. The plates will be planed all round.

Mountings.—The vessel will have the following mountings :—

One dome 30″ diameter × 30″ high of $\frac{5}{16}''$ plate with 6″ cast-steel stool.

One 16″ manhole frame and cover.

One 2″ deadweight safety valve of enclosed type and of approved make to blow off at 5 lbs. per square inch with cast-steel stool on dome.

One 6″ inlet pipe carried to bottom inside with 6″ valve and cast-steel stool.

One 6″ outlet pipe carried inside as shown with 6″ valve and cast-steel stool and supports.

One 6″ by-pass valve with 6″ wrought-iron or steel tees.

One 2″ cast-steel stool on end with 2″ gun-metal cock and wrought-iron or steel tee for run-off.

One 2″ cast-steel stool on bottom with 2″ gun-metal cock and wrought-iron or steel tee. This cast-steel stool will be riveted to a steel piece 12″ diameter × 12″ deep riveted to the shell.

Four 4″ wrought-iron or steel coils each in one piece, with flanges and jam nuts and supports as shown.

Eight cast-steel stools for ditto.

One set 8″ inlet connections, consisting of 8″ S bend and 8″ branch piece with four 4″ branches, all cast iron.

One set outlet connections, consisting of two 4″ × 6″ × 3″ special pieces and one 3″ special piece, all cast iron.

One set oil-level gauges of cast steel with glasses 1″ diameter, four spare glasses for each gauge.

Three steel plate and angle stools curved to fit vessel.

Dephlegmator.—The dephlegmator will be 3 feet diameter × 8 feet long, with two 8" cast-steel stools, one 3" cast-steel stool, one 15" manhole frame and door, and two angle lugs riveted on. The dephlegmator to be fitted with ¾" steam coil and steam valve.

One ¾" drain pipe to be provided for coil, carried to ground-level and there connected to a steam trap of the requisite capacity and of approved type.

Supporting Framing.—The supporting framing will be formed of steel H standards with cross-joists at top. The standards will be 10" × 6" H, with bases, tops, and brackets riveted on. The cross-joists at top will be 10" × 5", with knees riveted on where possible. The middle struts will be 8" × 5" H and 8" × 4" H, with knees and gussets riveted on where possible. There will be diagonal bracing on each transverse line of standards, and angle struts on each side of each transverse line for supporting the platform and dephlegmator.

The platform will be formed of 5/16" checkered plate bolted to 6" × 3" channels and angles, sufficient angles being provided to stiffen the plating. Holes are to be cut where pipes pass through. A handrailing is to be provided all round the platform with forged wrought-iron standards. The platform will be bolted throughout with ⅝" cup-headed bolts about 9" centres.

A stair is to be provided in two portions with middle landing. The stair to be formed of flat stringers with checkered plate treads. These treads will be stiffened on the outside edge.

SPECIFICATION OF RESIDUE PREHEATERS AND PLATFORM.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one set of residue preheaters and platform, all in accordance with this specification and fig. 55.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of this specification and contract.

The company will provide all foundations and will supply water for the test of the vessels. If the vessels require a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Residue Preheaters.—The set will consist of two complete heaters with top and bottom vessels. The lower vessel will be 10 feet diameter × 26 feet long, of 7/16" shell plates with ½" ends, all riveted with ¾" rivets at 2¼" pitch. The end plates will be stayed by gusset plates and angles. On one end there will be an opening for the insertion of the coils. This will be covered by a ⅝" plate riveted on.

The upper vessel will be 5 feet diameter × 26 feet long, of 5/16" plates riveted with ⅝" rivets 1¾" pitch.

These two vessels will be joined by two 3 feet diameter connections of 5/16" plate.

All rivet holes will be drilled in position through two or more thicknesses. After riveting, all seams will be caulked oil-tight against a pressure of 15 lbs. per square inch.

Each heater will have the following mountings:—

One dome 30" diameter × 30" high with 6" cast-steel stool, on upper vessel.

One 16" manhole frame and cover on upper vessel.

One 2″ deadweight safety valve of enclosed type and of approved pattern with cast-steel stool, on dome.

One 6″ wrought-iron inlet pipe carried to near bottom at centre with 6″ valve and cast-steel stool, on upper vessel.

One 6″ wrought-iron outlet pipe carried inside with 6″ valve and cast-steel stool and supports, on upper vessel.

One 6″ by-pass valve.

One 3″ cast-steel stool with 3″ cock and 3″ bend for lower vessel.

Three 4″ wrought-iron or steel coils each in one piece, with flanges, jam nuts, and supports as shown, in lower vessel.

Six cast-steel stools for ditto.

One set 6″ inlet, outlet, and by-pass connections of wrought-steel, with six 4″ valves and one 6″ valve, on lower vessel.

One 24″ manhole frame and cover on lower vessel.

One set cast-steel water-line gauges 1″ glass, four spare glasses for each, on upper vessel.

Cradles.—Cast-iron cradles are to be provided for the support of the lower vessels, three cradles for each vessel and each cradle in two pieces bolted together on the centre line.

Platforms.—One platform to be provided in front of lower vessel (see fig. 58), the platform to be 3 feet wide of $\frac{5}{16}$″ checkered plate and 3″ × 3″ × $\frac{3}{8}$″ angle steel.

A platform to be fitted to the front of the upper vessel, supported on brackets carried from the connecting branches between lower and upper vessels. A ladder will reach from ground-level to this upper platform. The ladder and upper platform will be fitted with handrailing on one side.

The two preheaters will be stayed to each other by diagonal bars of 3″ × 3″ × $\frac{3}{8}$″ angle steel.

SPECIFICATION OF PITCH STILL 10 FEET DIAMETER BY 25 FEET LONG INSIDE.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one still, 10 feet diameter × 25 feet long inside, all in accordance with this specification and fig. 59.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide the brickwork setting for the still, and will supply water for the test of the still. If the still requires a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Still.—The still will be 10 feet diameter × 25 feet long inside dimensions, and will be formed of $\frac{1}{2}$″ and $\frac{5}{8}$″ shell plates and $\frac{5}{8}$″ end plates. Each end plate will be in one piece and will be flanged for jointing to shell plates. The circular seams will be single riveted with $\frac{7}{8}$″ rivets at $2\frac{1}{2}$″ pitch, and the longitudinal seams double riveted with $\frac{7}{8}$″ rivets at 3″ pitch. All rivet holes will be drilled in position through two or more thicknesses.

The end plates will be stayed to the shell by four gusset stays of $\frac{3}{8}$″ plate and 3″ × 3″ × $\frac{3}{8}$″ angle steel for each end.

Mountings.—On the upper centre line of still there will be fitted one steel plate dome, 2 feet 6 inches diameter × 4 feet high of $\frac{3}{8}$″ plate, with one 3″ cast-steel stool and 3″ deadweight safety valve of closed pattern on top; this valve to blow at 5 lbs. per square inch and to be of design approved by engineers. This dome will also be fitted with one 8″ cast-steel stool.

One 24″ diameter steel manhole frame and cover will be fitted in position indicated on fig. 59. The flanges of frame and mandoor will be machined for jointing.

The still will be fitted with one 5″ cast-steel stool and 5″ valve for oil inlet.

The still will be fitted with one 3″ cast-steel stool and 3″ dipping cock.

The still will be fitted with one 3″ open steam pipe arranged as shown, with two lines of 2″ perforated pipe lying in bottom of still on angle bar supports. This 3″ pipe will enter the still through a special cast-steel stool, and will be fitted with one 3″ steam valve in the position shown.

The still will be fitted with one tapered wrought-steel stool 6½″ to 5″, of special design for run-off pipe. To this stool will be attached one 5″ gland cock, and this cock in turn will connect to the 6″ main run-down pipe later specified.

To the still there will be fitted eight cast-iron supporting brackets attached to the shell plates by special bolts tapped through the plates and secured by nuts on outside. Cast-iron bearing plates are to be provided for placing under these brackets.

SPECIFICATION OF STILL AND FITTINGS.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one steel still, 7 feet diameter × 19 feet long inside, all in accordance with this specification. (Continuous stills, fig. 51.)

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide the brickwork setting for the still, and will supply water for the first test of the still. If the still requires a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Still.—The still will be 7 feet diameter × 19 feet long inside dimensions, and will be formed of ½″ and ⅜″ shell plates and 7/16″ end plates. Each end plate will be in one piece and will be flanged for jointing to shell plates. The centre seam of the ½″ plates will be double riveted with ¾″ rivets at 2½″ pitch, all other seams will be single riveted with ¾″ rivets at 2″ pitch. All rivet holes will be drilled in position through two or more thicknesses.

After riveting all seams will be caulked oil-tight, and before delivery the still will be tested under water pressure of 20 lbs. per square inch.

The end plates will be stayed to the shell by four gusset stays of plates and angles for each end.

Mountings.—On the upper centre line of each still there will be fitted one cast-iron dome, 12 inches diameter × 2 feet 3 inches high, with one 2″ deadweight safety valve of closed pattern on top cover; this valve to blow at 5 lbs. per square inch. Design of safety valve to be approved.

One 18″ diameter steel manhole frame and cover will be fitted. The flanges of dome and manhole will be machined on flat jointing surfaces.

On the front plate of still there will be mounted one set of oil-level gauge fittings. These to be of cast steel with cocks and connecting pipes of 1″

fig. 137. As a rule receivers are erected in groups.

5000 Gallon Receiver.—This receiver will be 7 feet diameter × 23 feet long with dished ends. The plates of shell will be 5/16″ thick and for ends ⅜″, riveted with ⅝″ rivets at 1¾″ pitch.

The receiver will be riveted, caulked oil-tight, and tested with water against a pressure of 20 lbs. per square inch.

For the receiver there will be supplied two cast-iron cradles of the design and dimensions shown.

The receiver will be fitted with :—

One 24″ steel manhole frame and cover, machined on joints.
One 2″ cast-steel stool for dipping cock.
One 2″ dipping cock.
One 4″ cast-steel stool for inlet.
One 4″ inlet valve.
One steel facing for water run-off cock.
One 1¼″ water run-off cock.
One 5″ cast-steel bevelled stool for outlet.
One 5″ outlet valve.
One 4″ ventilator with nickel gauze screen.

Ladder and Gangway.—The receiver will be fitted with a gangway for access to the valves and pipes. This gangway will be 18 inches wide of ¼″ checkered plate and 2½″ × 2½″ × ⅜″ angle steel runners. Access from ground-level will be provided at one end by a steel ladder 18 inches wide, as shown on fig. 137. As a rule receivers are erected in groups.

SPECIFICATION OF CONDENSER TANK 12 FEET LONG BY 8 FEET WIDE BY 7 FEET 6 INCHES DEEP, WITH RADIATOR PIPES.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one condenser tank 12 feet long × 8 feet wide × 7 feet 6 inches deep, all in accordance with this specification and fig. 69.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide all foundations.

Condenser.—The condenser tank will measure 12 feet long × 8 feet wide × 7 feet 6 inches deep, and will be formed of ¼″ steel plates and 2½″ × 2½″ × ⅜″ angle steel, with cross and longitudinal stays 2½″ × ⅜″ flat steel attached to vertical angle steel stiffeners of 2½″ × 2½″ × ⅜″ section.

The rivet holes in these plates and bars may be punched, but when erected must be quite fair and in good line. All plates and angles must be planed for caulking and corners thinned where necessary. Angle joints to be carefully butted and covered with angles not less than 18″ in length.

For the condenser tank there will be required 192 radiator pipes of the form and dimensions indicated on fig. 69. The flanges on these pipes must be machined so that each pipe will measure exactly 5½″ across. The pipes in condenser are arranged in two sections, each in eight vertical lines of twelve pipes, and these lines are connected on top to the vapour pipes from preheaters or stills. At the bottom each section is connected to a seal vessel which is connected to the run-down pipes.

The radiator pipes are to be supported from bottom of condenser tank on steel channel bars, the lower bar in each case being riveted to tank bottom plates as a stiffener.

Fittings.—The tank is to be fitted with one 4″ cast-iron overflow pipe to ground-level, and with one 3″ cast-iron sludge cock, and cast-iron connection to ground-level.

The water inlet pipe will be 3″ diameter with gun-metal gland cock and 3″ pipe to bottom of tank. This 3″ pipe will be connected to the main pipe on top of tank.

One gangway 2 feet wide will be provided on the top of the tank with ladder from ground-level. The gangway will be constructed of $\frac{5}{16}$″ checkered plates carried on 3″ × 3″ × ⅜″ angle steel runners.

The ladder will be constructed of 4″ × ⅜″ flat bars and $\frac{5}{16}$″ checkered plate treads, stiffened on outer edge by a flat bar riveted to each step. Care to be taken that these treads are arranged for easy ascent and descent.

Single handrail will be provided along the gangway and double handrails on the ladder. These handrails to be of 1″ bore tube with 1″ diameter solid standards.

Testing.—After erection the tank will be tested by being filled with water to the top and left for at least twenty-four hours. It must be perfectly tight. After the tank has been satisfactorily tested each set of radiator pipes will be tested under a head of 10 feet and left standing for twenty-four hours. There must be no leaks.

Note.—Almost without exception these condenser tanks are arranged in groups, in which case one ladder would be provided for the group and the gangway would be fitted across the complete set of tanks.

SPECIFICATION OF CONDENSER TANK 12 FEET LONG BY 8 FEET WIDE BY 9 FEET DEEP, WITH COIL PIPES.

Extent of Contract.—This contract will include the supply, delivery, erection, and testing of one condenser tank, 12 feet long × 8 feet wide × 9 feet deep, all in accordance with this specification and fig. 68.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide all foundations.

Condenser.—The condenser tank will measure 12 feet long × 8 feet wide × 9 feet deep, and will be formed of ¼″ steel plates and 2½″ × 2½″ × ⅜″ angle steel, with longitudinal stays of 2½″ × ⅜″ flat steel attached to vertical angle steel stiffeners of 2½″ × 2½″ × ⅜″ sections. The cross-stays to be of flat iron combined with 5″ × 3″ H joists placed vertically. Along the bottom two 5″ × 3″ H joists will be fitted and riveted to bottom plates.

The rivet holes in these plates and bars may be punched, but when erected must be quite fair and in good line. All plates and angles must be planed for caulking and corners thinned where necessary. Angle joints to be carefully butted and covered with angles not less than 18″ in length.

For the condenser tank there will be supplied and fitted two complete coils of 4″ cast-iron pipes. Each coil will include straight pipes and return bends of light pattern for lower end and straight pipes and return bends of heavy pattern for inlet to coil. All sockets on these pipes must be 7″ deep. The inlet to each coil will consist of one cast-iron taper pipe, 6″ to 4″, and of two 6″ cast-iron bends. One 6″ × 8″ cast-iron tee-piece will connect the last-mentioned bends.

The worm end of each coil will connect out through the condenser tank into one vacuum vessel and into one seal vessel. These seal vessels are connected by two special 3″ branch pipes to four 3″ cocks controlling the four lines of 4″ tail pipes which again connect to receivers.

Condenser coils are to be supported from bottom of tank by four cast-iron blocks of required heights for each coil.

Fittings.—The tank is to be fitted with one 4″ wrought-iron overflow pipe to ground-level and with one 3″ cast-iron cock and 3″ sludge pipe to ground-level.

The water inlet pipe will be 3″ diameter with gun-metal gland cock and 3″ pipe to bottom of tank. This 3″ pipe will be connected to the main pipe on top of tank.

One gangway 2 feet wide will be provided on the top of the tank with ladder from ground-level. The gangway will be constructed of $\frac{5}{16}''$ checkered plates carried on $3'' \times 3'' \times \frac{3}{8}''$ angle steel runners.

The ladder will be constructed of $4'' \times \frac{3}{8}''$ flat bars and $\frac{5}{16}''$ checkered plate treads stiffened on outer edge by a flat bar riveted to each step. Care to be taken that these treads are arranged for easy ascent and descent.

Single handrail will be provided along the gangway and double handrails on the ladder. These handrails to be of 1″ bore tube with 1″ diameter solid standards.

Testing.—After erection the tank will be tested by being filled with water to the top and left for at least twenty-four hours. It must be perfectly tight. After the tank has been satisfactorily tested each coil will be tested under a head of 10 feet and left standing for twenty-four hours. There must be no leaks.

Note.—Almost without exception these condenser tanks are arranged in groups, in which case one ladder would be provided for the group and the gangway would be fitted across the complete set of tanks.

SPECIFICATION OF 20,000-GALLON VERTICAL OIL WASHER, WITH MOUNTINGS.

Extent of Contract.—The contract will include the supply, delivery, erection, and testing of one steel washer, 16 feet 6 inches diameter × 16 feet deep, all in accordance with this specification and fig. 138.

The contract will be subject to all the conditions and terms laid down in the general conditions issued by the purchasers, and it must be understood that these general conditions form part of the specification and contract.

The company will provide the foundation under the washer, and will supply water for the test of the washer. If the washer requires a second filling on test, the contractor will meet the cost of this or any subsequent refilling.

Washer.—The washer will be 16 feet 6 inches diameter × 16 feet deep on the cylindrical portion, with a conical portion 5 feet 6 inches deep to the point, and a domed cover rising 12″ above the cylindrical part of the vessel.

The cylindrical shell will be formed of $\frac{3}{8}''$ steel plates and the conical part of $\frac{1}{2}''$ plates riveted with $\frac{3}{4}''$ rivets at $2\frac{1}{4}''$ pitch. The upper edge of the shell will finish with an angle steel curb $3'' \times 3'' \times \frac{3}{8}''$ in section riveted to shell by $\frac{3}{4}''$ rivets $4\frac{1}{2}''$ pitch. One ring of angle steel $3'' \times 3'' \times \frac{3}{8}''$ in section will surround the shell and will be riveted to the plates with $\frac{3}{4}''$ rivets at 6″ pitch.

per cock a connection will be made to the oil run-off line.
ecessary pipe supports are to be included.

APPENDIX.

A LIST OF REFERENCES USEFUL TO THE PETROLEUM REFINER

to be found in literature in the English language.

WHERE identification letters and page numbers are necessary they are printed in italics.

The list is arranged under subject headings not included in the subject index. Author's names are included in the name index.

Journals and Reference Letters.

JOURNAL OF THE SOCIETY OF CHEMICAL INDUSTRY.

Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter
1	*A*	10	*J*	19	*S*	28	*b*
2	*B*	11	*K*	20	*T*	29	*c*
3	*C*	12	*L*	21	*U*	30	*d*
4	*D*	13	*M*	22	*V*	31	*e*
5	*E*	14	*N*	23	*W*	32	*f*
6	*F*	15	*O*	24	*X*	33	*g*
7	*G*	16	*P*	25	*Y*	34	*h*
8	*H*	17	*Q*	26	*Z*	35	*i*
9	*I*	18	*R*	27	*a*	36	*ia*

JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY.

Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter.
1	*q*	4	*n*	7	*k*
2	*p*	5	*m*	8	*j*
3	*o*	6	*l*	9	*ja*

JOURNAL OF INSTITUTION OF PETROLEUM TECHNOLOGISTS.

Vol.	Reference letter.
2	*r*
3	*ra*

PETROLEUM REVIEW.

Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter.
1	*s*	11	*D/*	21	*N/*	31	*X/*
2	*t*	12	*E/*	22	*O/*	32	*Y/*
3	*u*	13	*F/*	23	*P/*	33	*Z/*
4	*v*	14	*G/*	24	*Q/*	34	*a/*
5	*x*	15	*H/*	25	*R/*	35	*b/*
6	*y*	16	*I/*	26	*S/*	36	*b/a*
7	*z*	17	*J/*	27	*T/*	37	*b/b*
8	*A/*	18	*K/*	28	*U/*		
9	*B/*	19	*L/*	29	*V/*		
10	*C/*	20	*M/*	30	*W/*		

PETROLEUM WORLD.

Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter.	Vol.	Reference letter.
1	*c/*	5	*g/*	9	*k/*	13	*o/*
2	*d/*	6	*h/*	10	*l/*	14	*o/a*
3	*e/*	7	*i/*	11	*m/*		
4	*f/*	8	*j/*	12	*n/*		

Abbreviations.

The following are the abbreviations employed to indicate a large number of the publications in which the articles referred to appeared :—

Abh. der Bakuer Abt. Kaiserl. niss. tech. Ges.	*See* Trudy bak.
Allgem. Oesterr. Chem. t. Zeit.	Allgemeine Oesterreichische Chemisch technische Zeitung.
Amer. Chem. Journ. . .	American Chemical Journal.
Amer. Soc. Mech. Eng. .	American Society of Mechanical Engineers.
Ann.	Annalen der Chemie.
Ann. Accad. Scient. Fennicæ	Ann. Accademie Scientiarum Fennicæ.
Annali Chim. Appl. . .	Annali di Chimica Applicata.
Ann. Chim. Phys. . .	Annales de Chimie et de Physique.
Atti R. Accad. Linc. . .	Atti della Reale Accademia dei Lincei.
Ber.	Berichte der Deutschen Chemischen Gesellschaft.
Bull. Soc. Chim. . . .	Bulletin de la Société chimique de France.
Bull. Soc. de Stiinte din Bucuresti	Bulletin de la Societa de Stiinte din Bucuresti.
Bull. Soc. d'Encour. . .	Bulletin de la Société d'Encouragement du Nord de la France.
Bull. Soc. Mulhouse . .	Bulletin de la Société Industrielle de Mulhouse.
Chem. and Drug. . . .	Chemist and Druggist.
Chem. Centr. (or *Zentr.*) .	Chemisches Zentralblatt.
Chem. Ind.	Chemische Industrie.

Chem. Rev. Harz Ind.	Chemische Revue über die Fett- und Harz Industrie.
Chem. Soc. Trans.	Transactions of the Chemical Society.
Chem. Tech. Rev.	Chemisch-Technische Revue.
Chem. Trades Jour.	Chemical Trades Journal.
Chem. Umschau	Chemisches Umschau.
Chem. Zeit.	Chemiker Zeitung.
Compt. Rend.	Comptes Rendus hebdomadaires des Séances de l'Académie des Sciences.
Concr. and Constr. Eng.	Concrete and Constructional Engineering.
Ding. Poly.	Dingler's polytechnisches Journal.
Eng. Record	Engineering Record.
Eng. and Min. J.	Engineering and Mining Journal.
Färb. Zeit.	Färber Zeitung.
Gorny Journ.	Mining Journal (Russ.), Petrograd.
Gummi-Zeit.	Gummi-Zeitung.
J. Agric. Res.	Journal of Agricultural Research.
J. Amer. Chem. Soc.	Journal of the American Chemical Society.
J. Frank. Inst.	Journal of the Franklin Institute.
J. Gasbel.	Journal für Gasbeleuchtung und Wasserversorgung.
J. Pharm. Chim.	Journal de Pharmacie et de Chimie.
J. Russ. Phys. Chem.	Journal of the Physical and Chemical Society of Russia.
J. Phys. Chem.	Journal of Physical Chemistry.
J. prakt. Chem.	Journal für praktische Chemie.
J. Soc. Arts	Journal of the Royal Society of Arts.
Karlsrüher Chem. Ges.	Karlsrüher Chemische Gesellschaft.
Koll. Zeits.	Kolloid Zeitschrift.
Les Mat. Gras.	Les Matières Grasses.
Mem. der Kais. Russ. t. Ges.	*See* Zap. imp. Russk.
Met. and Chem. Eng.	Metallurgical and Chemical Engineering.
Min. and Scient. Press	Mining and Scientific Press.
Mitt. k. Mat.	Mittheilungen aus dem königlichen Materialprüfungsamt zü Gross-Lichterfelde West.
Mitt. k. t. Gew.-mus. in Wien	Mittheilungen des k. technischen Gewerbemuseums in Wien.
Mitt. k. t. Ver.	Mittheilungen des k.k. technischen Versuchsamtes.
Monatsh. Chem.	Monatshefte für Chemie der kaiserlichen Akademie der Wissenschaften, Wien.
Monit. Scient.	Moniteur Scientifique.
Neftj. Djelo	Neftannoie Dielo. (Oil trade.)
Oesterr. Berg. Hüt.	Oesterreichische Zeitschrift für Berg und Hüttenwesen.
Oesterr. Chem.-t. Zeit.	Oesterreichische Chemisch-technische Zeitung.
O. and C. T. J.	Oil and Colour Trade Journal.
O. P. and D. R.	Oil, Paint, and Drug Reporter.
P.	Petroleum.
P. Ind. Techn. Rev.	Original name of " Petroleum Review."
Pharm. Zeit.	Pharmazeutische Zeitung.

Phot. Corr.	Photographische Correspondenzblatt.
Proc. Amer. Phil. Soc.	Proceedings of the American Philosophical Society, Philadelphia.
Proc. Inst. Civ. Eng.	Proceedings of the Institution of Civil Engineers.
Rev. de Chim. Ind.	Revue de Chimie Industrielle.
Rev. de Pétrole	Revue de Pétrole.
Rev. Prod. Chim.	Revue des Produits Chimiques.
Scient. Amer.	Scientific American.
Scient. Amer. Suppl.	Scientific American, Supplement.
Tech. West.	Technical Journal (Russ.).
Trudy bak.	Proceedings of the Baku Branch of the Imperial Russian Technical Society.
U.S. Bull. Bur. Standards	United States Bulletin, Bureau of Standards.
West. shir. Wes.	Journal of Fatty Matters (Russ.).
Zap. Imp. Russk.	Journal of the Imperial Russian Technical Society.
Z. anal. Chem.	Zeitschrift für analytischen Chemie.
Z. ang. Chem.	Zeitschrift für angewandte Chemie.
Z. Elektrochem.	Zeitschrift für Elektrochemie.
Zeits. für Spir.	Zeitschrift für Spiritusindustrie.
Z. ges. Brauw.	Zeitschrift für das gesammte Brauwesen.
Zeits. ö. Chem.	Zeitschrift für öffentliche Chemie.
Z. phys. Chem.	Zeitschrift für physikalische Chemie.
Z. für prakt. Geol. Chem.	Zeitschrift für praktische Geologie und Chemie.

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